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18 March 1953
Report No. 682
(Quarterly)
Copy No.

EXPLOSIVES RESEARCH

Contract N7onr-46208

18 March 1953

Report No. 682
(Quarterly)

EXPLOSIVES RESEARCH

Contract N7onr-46208

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CONTENTS

CONTENTS (cont.)

CONTRACT FULFILLMENT STATEMENT

This quarterly report is submitted in partial fulfillment of Contract N7onr-46208.

I. SUMMARY

A. This quarterly report is submitted under Contract N7onr-46208 and covers the period 1 November 1952 through 31 January 1953.* The objectives of the contract are threefold:

1. Synthesis and physical studies of new high-energy polynitro compounds as potential explosives.

2. Desensitization of RDX with materials which will not lower its oxygen balance appreciably.

3. Development of bomb and shell casings made of an explosive plastic.

B. The more important results and conclusions of the work reported are presented below:

1. Four new aliphatic polynitronitramines have been synthesized by use of the Mannich condensation.

a. 3,3,5,7,7-Pentanitro-5-aza-nonane and 4,4,6,8,8-pentanitro-6-aza-undecane were prepared by the condensation of ammonium acetate with 2,2-dinitrobutanol and 2,2-dinitropentanol, respectively, followed by postnitration.

b. The condensation of 3,3-dinitro-1,5-pentanediamine with two moles of trinitroethanol, followed by postnitration, gave 1,1,1,3,6,6,9,11,11,11-decanitro-3,9-diaza-undecane. This compound has a calculated ballistic-mortar value of 155 (RDX = 150) and a calculated lead-block value of 177 (RDX = 157).

c. The condensation of methylene diamine with two moles of trinitroethanol, followed by postnitration, gave 1,1,1,3,5,7,7,7-octanitro-3,5-diaza-heptane. This compound has an oxygen balance of +10.4, and when adjusted to zero oxygen balance with 12.3% TNT, it has a calculated ballistic-mortar value of 156.3 and a calculated lead-block value of 195.5.

2. 3,3,3-Trinitropropyl amine nitrate was prepared by treating 3,3,3-trinitropropyl isocyanate with dilute nitric acid. This salt has a calculated ballistic-mortar value of 144 and a calculated lead-block value of 171.

3. Two new heterocyclic polynitro compounds have been synthesized:

a. 1,3,5-tris(3',3',3'-Trinitropropyl-hexahydro-1,3,5-triazine was prepared by the condensation of three moles of 3,3,3-trinitropropyl amine with three moles of formaldehyde.

*Previous work on this contract was covered in Aerojet Reports No. 512, 538, 562, 589, 621, 637, and 660.

b. By the condensation of 1,1,1,6,6,11,11,11-octanitro-4,8-diaza-undecane and formaldehyde, a compound was obtained which was postulated to be 1,3-bis(3',3',3'-trinitropropyl)-5,5-dinitro-hexahydro-1,3-diazine. This compound has a calculated ballistic-mortar value of 157 and a calculated lead-block value of 153.

4. The following samples were prepared and submitted to the Naval Ordnance Laboratory for evaluation:

a. 1,1,1,4,6,6,8,11,11,11-Decanitro-4,8-diaza-undecane

b. 1,1,1,3,6,6,9,11,11,11-Decanitro-3,9-diaza-undecane

c. 1,1,1,3,5,7,7,7-Octanitro-3,5-diaza-heptane

d. 1,1,1,3,6,6,6-Heptanitro-3-aza-hexane

e. N-3',3',3'-Trinitropropyl-3,3,5,5-tetranitropiperidine

f. 3,3,3-Trinitropropyl amine nitrate

g. 3,3,5,7,7-Pentanitro-5-aza-nonane.

The evaluation of six of these compounds, together with three compounds submitted last quarter, has been completed and is included in this report.

5. The investigation of new surface-active agents for use in desensitizing RDX has been continued. Values of hydrophile-lipophile balance (HLB) were compared with percents adsorbed on RDX from acetone-water for a large number of wetting agents supplied by the Atlas Powder Company. It has been found that high adsorption correlates fairly well with low HLB. Some of these new Atlas wetting agents show promise for use in the desensitization of RDX.

II. TECHNICAL PROGRESS: SYNTHESIS OF NEW HIGH EXPLOSIVES

A. INTRODUCTION

1. The present explosives program is directed toward the synthesis of new, stable, high-energy polynitro compounds with a preferred oxygen balance on the positive side or very near zero on the negative side.

2. This report deals mainly with the synthesis of aliphatic polynitronitramines and heterocyclic polynitro compounds.

B. PREPARATION OF ALIPHATIC POLYNITRONITRAMINES

1. Preparation of 3,3,5,7,7-Pentanitro-5-aza-nonane and 4,4,6,8,8-Pentanitro-6-aza-undecane

a. Discussion

(1) The preparation of 2,2,4,6,6-pentanitro-4-aza-heptane by the Mannich condensation of 2,2-dinitropropanol and ammonium acetate, followed by postnitration, has been previously reported.* This reaction has now been extended to the synthesis of the next higher homologs, 3,3,5,7,7-pentanitro-5-aza-nonane and 4,4,6,8,8-pentanitro-6-aza-undecane. It was hoped that by increasing the carbon chain in this manner a waxy material might be obtained which would be suitable for the coating of RDX. However, these new nitramines are not waxy, but crystalline.

(2) Two moles of 2,2-dinitrobutanol condensed readily with ammonium acetate to form 3,3,7,7-tetranitro-5-aza-nonane (Ia), a white crystalline compound, mp 66 to 67°C, I.S. $>$100 cm/2 kg. Nitration of (Ia) with a mixture of 100% nitric acid and acetic anhydride gave 3,3,5,7,7-pentanitro-5-aza-nonane (IIa), a white crystalline compound, mp 106 to 107°C, I.S. $>$100 cm/2 kg.

(3) Similarly, two moles of 2,2-dinitropentanol condensed with ammonium acetate to form 4,4,8,8-tetranitro-6-aza-undecane (Ib), a white crystalline compound, mp 100 to 100.5°C. Nitration of (Ib) with a mixture of 100% nitric acid and acetic anhydride gave 4,4,6,8,8-pentanitro-6-aza-undecane (IIb), a white crystalline compound, mp 101 to 103°C.

$$2\,R\overset{NO_2}{\underset{NO_2}{C}}CH_2OH + NH_4OCOCH_3 \longrightarrow \left[R\overset{NO_2}{\underset{NO_2}{C}}CH_2\right]_2\!-NH \xrightarrow[Ac_2O]{HNO_3} \left[R\overset{NO_2}{\underset{NO_2}{C}}CH_2\right]_2 NNO_2$$

(Ia), R = CH_3CH_2 (IIa), R = CH_3CH_2

(Ib), R = $CH_3CH_2CH_2$ (IIb), R = $CH_3CH_2CH_2$

b. Experimental

(1) Preparation of 3,3,7,7-Tetranitro-5-aza-nonane

(a) A mixture of 30.0 g (0.183 mole) of 2,2-dinitrobutanol, 50 ml of water, and 30.0 g (0.39 mole) of ammonium acetate

*Aerojet Report No. 621, p. 2.

was heated on the steam bath for 20 minutes. The reaction mixture was cooled, and the product was collected and dried, giving 28.0 g (99%) of a cream-colored solid, mp 57 to 61°C. Recrystallization from isopropyl alcohol gave white plates, mp 66 to 67°C, I.S. >100 cm/2 kg.

Anal. Calc'd for $C_8H_{15}N_5O_8$: %C, 31.07; %H, 4.89; %N, 22.65

Found: %C, 31.06; %H, 4.98; %N, 21.73

(2) Preparation of 3,3,5,7,7-Pentanitro-5-aza-nonane

(a) A mixture of 100 ml of acetic anhydride and 150 ml of 100% nitric acid was cooled to 5°C, and 18 g (0.0582 mole) of 3,3,7,7-tetranitro-5-aza-nonane was added. The solution was poured onto ice to give a white solid. The product was collected, washed with water, and dried _in vacuo_ over potassium hydroxide. The yield was quantitative, mp 97 to 99°C. Recrystallization from chloroform gave white plates, mp 106 to 107°C, I.S. >100 cm/2 kg.

Anal. Calc'd for $C_8H_{14}N_6O_{10}$: %C, 27.12; %H, 3.98; %N, 23.73

Found: %C, 27.17; %H, 3.72; %N, 22.74

Heat of Combustion, ΔH_p, Predicted: 3290 cal/g

Found: 3234 cal/g

(3) Preparation of 2,2-Dinitropentanol

(a) In a 200-ml 3-necked flask, fitted with a mechanical stirrer, thermometer, and dropping funnel, was placed 14.8 g (0.1 mole) of 1,1-dinitrobutane and 45 ml of water. A solution of 4.2 g (0.1 mole) of 95% sodium hydroxide in 15 ml of water was added dropwise, keeping the temperature at 25°C by external cooling. A quantity of 8.1 g (0.1 mole) of 37% formalin was added dropwise, the reaction mixture was stirred for 45 minutes, and a solution of 5.1 g (0.05 mole) of concentrated sulfuric acid in 10 ml of water was added dropwise at 25°C. The solution was extracted with methylene chloride, and the extract was dried and concentrated to give 11.6 g of oil.

(4) Preparation of 4,4,8,8-Tetranitro-6-aza-undecane

(a) A quantity of 11.6 g of 2,2-dinitropentanol was warmed on the steam bath for 20 minutes with a solution of 11.6 g of ammonium acetate in 100 ml of water. On cooling a solid precipitated, which was collected, washed with water, and dried to give 6.2 g of a yellow solid, mp 81 to 85°C. Recrystallization from ethanol gave white rods, mp 100 to 100.5°C.

Anal. Calc'd for $C_{10}H_{19}N_5O_8$: %C, 35.61; %H, 5.68; %N, 20.77

Found: %C, 35.78; %H, 5.58; %N, 19.75

(5) Preparation of 4,4,6,8,8-Pentanitro-6-aza-undecane

(a) To a solution of 100 ml of 100% nitric acid and 100 ml of acetic anhydride, cooled to 5°C, was added 6.2 g of crude 4,4,8,8-tetranitro-6-aza-undecane. The solution was poured on ice and a white solid precipitated. The product was collected, washed with water, and dried to give 5.8 g of white solid, mp 65 to 70°C. Two recrystallizations from carbon tetrachloride raised the melting point to 101 to 103°C.

Anal. Calc'd for $C_{10}H_{18}N_6O_{10}$: %C, 31.42; %H, 4.75; %N, 21.99

Found: %C, 31.70; %H, 4.85; %N, 21.22

2. Purification of 1,1,1,4,6,6,8,11,11,11-Decanitro-4,8-diaza-undecane

a. Discussion

(1) The preparation of 1,1,1,4,6,6,8,11,11,11-deca-nitro-4,8-diaza-undecane (III) has been previously reported.* This compound was purified by recrystallization from hot concentrated nitric acid; the mp was 135 to 137°C dec. It has now been found that a purer compound, mp 157 to 159°C dec., with a better analysis can be obtained by recrystallization at room temperature from 100% nitric acid and water, or methanol and water.

b. Experimental

(1) 1,1,1,4,6,6,8,11,11,11-Decanitro-4,8-diaza-undecane was purified by dissolving in 100% distilled nitric acid at room temperature and adding just enough water to cause precipitation. This procedure gave a white crystalline solid, I.S. = 5 to 10 cm/2 kg, mp 157 to 159°C dec. A similar purification from methanol and water did not raise the melting point.

Anal. Calc'd for $C_9H_{12}N_{12}O_{20}$: %C, 17.77; %H, 1.99; %N, 27.64

Found: %C, 18.29; %H, 2.02; %N, 27.40

Heat of Combustion, ΔH_p, Predicted: 2044 cal/g

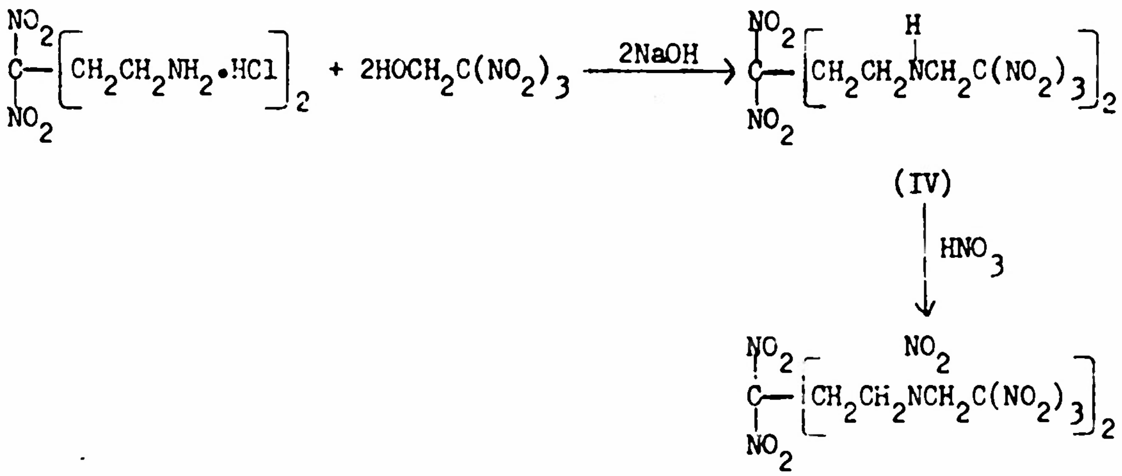
Found: 1982, 1984 cal/g

3. Preparation of 1,1,1,3,6,6,9,11,11,11-Decanitro-3,9-diaza-undecane

a. Discussion

(1) The Mannich condensation of 3,3-dinitro-1,5-pentanediamine with two moles of trinitroethanol gave 1,1,1,6,6,11,11,11-octanitro-3,9-diaza-undecane (IV). Nitration of (IV) with a mixture of 100% nitric acid and acetic anhydride gave 1,1,1,3,6,6,9,11,11,11-decanitro-3,9-diaza-undecane (V).

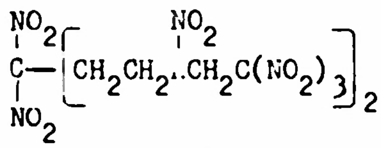
*Aerojet Report No. 660



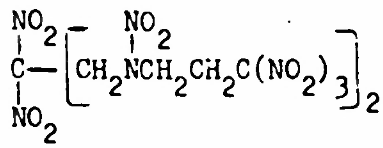
(V)

(V) is a white crystalline compound, mp 170 to 175°C dec., I.S. = 10 to 15 cm/2 kg, with a calculated lead-block value of 177 and a calculated ballistic-mortar value of 155.

(2) Compound (V) is isomeric with 1,1,1,4,6,6,8,11,11,11-decanitro-4,8-diaza-undecane (III):



(V)



(III)

This preparation provides another means* for comparing the properties of the N-nitro-N-trinitroethyl grouping with those of the N-nitro-N-trinitropropyl grouping.

b. Experimental

(1) Preparation of 1,1,1,6,6,11,11,11-Octanitro-3,9-diaza-undecane

(a) A solution of 26.5 g (0.1 mole) of 3,3-dinitro-1,5-pentanediamine dihydrochloride, 36.2 g (0.2 mole) of trinitroethanol, and 100 ml of water was placed in a 500-ml 3-necked flask, fitted with a mechanical stirrer. A solution of 41 ml of 4.8777N sodium hydroxide (0.2 mole) was added dropwise from a burette. The addition of

*See also Aerojet Report No. 660, p. 8.

the base caused a yellow solid to precipitate. The product was collected, washed with water, and dried to give 34 g of yellow solid, mp 93 to 95°C dec.

(2) Preparation of 1,1,1,3,6,6,9,11,11,11-Decanitro-3,9-diaza-undecane

(a) A solution of 250 ml of 100% nitric acid and 250 ml of acetic anhydride was cooled to 5°C, and 34 g of 1,1,1,6,6,11,11,11-octanitro-3,9-diaza-undecane was added. The solution was poured on ice, causing a white solid to precipitate. The product was collected, washed with water, and dried to yield 27.8 g, mp 110 to 114°C dec. Two recrystallizations from hot 100% distilled nitric acid raised the melting point to 170 to 175°C dec., I.S. = 10 to 15 cm/2 kg.

Anal. Calc'd for $C_9H_{12}N_{12}O_{20}$:		%C, 17.77; %H, 1.99; %N, 27.64
	Found:	%C, 18.11; %H, 2.00; %N, 27.34
Heat of Combustion, ΔH_p,	Predicted:	2044 cal/g
	Found:	1956, 1978 cal/g

4. Preparation of 1,1,1,3,5,7,7,7-Octanitro-3,5-diaza-heptane

a. Discussion

(1) The condensation of 3,3,3-trinitropropyl amine with trinitroethanol, 2,2-dinitro-1,3-propanediol, and 2,2,4,4-tetranitro-1,5-pentanediol has been described previously.* The present phase of the work is a study of the condensation of methylene diamine with these polynitro-alcohols.

(2) It has been found that methylene diamine will condense in an anhydrous medium with two moles of trinitroethanol to give 1,1,1,7,7,7-hexanitro-3,5-diaza-heptane (VI), a bright-yellow solid, mp 100 to 105°C dec. Nitration of (VI) with a mixture of 100% nitric acid and acetic anhydride gave 1,1,1,3,5,7,7,7-octanitro-3,5-diaza-heptane (VII):

$$CH_2(NH_2\cdot HCl)_2 + 2HOCH_2C(NO_2)_3 + 2NaOCOCH_3$$

$$\downarrow$$

$$CH_2\left[\overset{\displaystyle H}{\overset{|}{N}}CH_2C(NO_2)_3\right]_2$$

(VI)

$$\Big\downarrow\begin{matrix}HNO_3\\Ac_2O\end{matrix}$$

$$CH_2\left[\overset{\displaystyle NO_2}{\overset{|}{N}}CH_2C(NO_2)_3\right]_2$$

(VII)

*Aerojet Report No. 660, pp. 3-7.

(VII) is a white crystalline compound, mp 84 to 85°C, I.S. = 5 to 10 cm/2 kg, with an oxygen balance of +10.4. When adjusted to zero oxygen balance with 12.3% TNT, (VII) has a calculated ballistic-mortar value of 156 and a calculated lead-block value of 196.

(3) The condensation of methylene diamine with bifunctional alcohols such as 2,2-dinitro-1,3-propanediol and 2,2,4,4-tetranitro-1,5-pentanediol gave gummy products, indicating that polymerization took precedence over the formation of the desired cyclized compounds. For the present such compounds will not be investigated further, but the investigation may be resumed if new methods of conducting such reactions are discovered.

b. Experimental

(1) Preparation of Methylene Diamine Dihydrochloride*

(a) Methylene diformamide, 63.6 g (0.623 mole), was dissolved in the minimum amount of concentrated hydrochloric acid at 12°C to give a clear solution. After the solution was permitted to stand overnight, a white crystalline solid separated, which was collected and dried *in vacuo* over potassium hydroxide, yielding 31.1 g (41.8%), mp 230 to 240°C dec.

(2) Preparation of 1,1,1,7,7,7-Hexanitro-3,5-diaza-heptane

(a) In a one-liter 3-necked flask, fitted with a sealed mechanical stirrer, reflux condenser with drying tube, and dropping funnel, was placed 21.0 g (0.176 mole) of methylene diamine dihydrochloride and 600 ml of methanol. On refluxing, a clear solution was obtained. The solution was cooled to room temperature, and 63.9 g (0.352 mole) of trinitroethanol was added. A solution of 28.9 g (0.352 mole) of anhydrous sodium acetate in 300 ml of methanol was added dropwise. The yellow solution was allowed to stand overnight and concentrated *in vacuo* at 40°C. The yellow solid residue was extracted with absolute ethanol and acetone, leaving 20.4 g of sodium chloride (theoretical = 20.6 g). The extracts were concentrated *in vacuo*, leaving 61.0 g (93.1%) of yellow solid, mp 100 to 105°C dec., I.S. = 40 cm/2 kg.

(3) Preparation of 1,1,1,3,5,7,7,7-Octanitro-3,5-diaza-heptane

(a) A nitrating mixture of 250 ml of 100% nitric acid and 250 ml of acetic anhydride was cooled to 5°C, and 48.8 g (0.131 mole) of 1,1,1,7,7,7-hexanitro-3,5-diaza-heptane was added. The solution was stirred for 15 minutes and poured onto ice. A white solid separated in about one hour; it was collected, washed with water, and dried *in vacuo* over potassium hydroxide, yielding 14.8 g (24.4%), mp 75 to 79°C. Recrystallization from cyclohexane raised the melting point to 84 to 85°C, I.S. = 5 to 10 cm/2 kg.

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*Knudsen, Ber. 47, 2699 (1914).

Anal. Calc'd for $C_5H_6N_{10}O_{16}$: %C, 12.99; %H, 1.31; %N, 30.31

Found: %C, 13.44; %H, 1.23; %N, 30.45

Heat of Combustion, ΔH_p, Predicted: 1427 cal/g

Found: 1423 cal/g

C. PREPARATION OF 3,3,3-TRINITROPROPYL AMINE NITRATE

1. Discussion

a. 3,3,3-Trinitropropyl amine hydrochloride has been prepared by treating 3,3,3-trinitropropyl isocyanate with concentrated hydrochloric acid.* Because of its favorable oxygen balance it was of interest to synthesize the corresponding nitrate salt and evaluate its physical properties.

b. 3,3,3-Trinitropropyl amine nitrate was prepared by treating 3,3,3-trinitropropyl isocyanate with 35% nitric acid. The nitrate salt is a white crystalline compound, mp 130 to 133°C dec., I.S. = 90 cm/2 kg, with a calculated ballistic-mortar value of 143.7 and a calculated lead-block value of 170.9.

2. Experimental

a. A mixture of 6.6 g (0.03 mole) of 3,3,3-trinitropropyl isocyanate with 50 ml of 35% nitric acid was heated on the steam bath for three hours. The solution was concentrated *in vacuo* to give a white solid. The product was collected, washed with ether, and dried, yielding 6.6 g (85.7%), mp 130 to 133°C dec., I.S. = 90 cm/2 kg. Recrystallization from a mixture of ethyl acetate and ether did not raise the melting point.

Anal. Calc'd for $C_3H_7N_5O_9$: %C, 14.01; %H, 2.74; %N, 27.24

Found: %C, 14.09; %H, 2.80; %N, 26.58

Heat of Combustion, ΔH_p, Predicted: 1706 cal/g

Found: 1654, 1659 cal/g

D. PREPARATION OF HETEROCYCLIC POLYNITRO COMPOUNDS

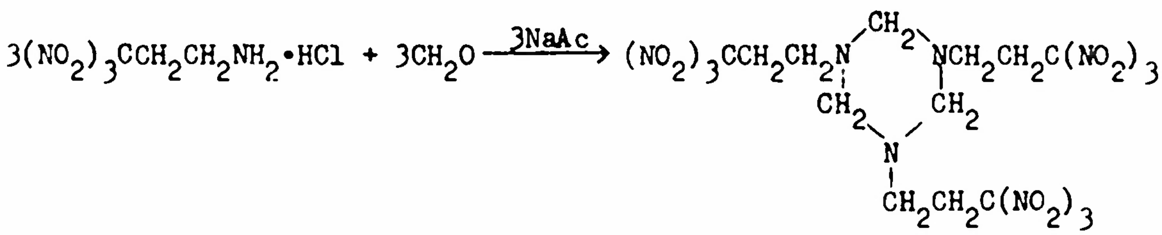
1. Preparation of 1,3,5-tris(3',3',3'-Trinitropropyl)-hexahydro-1,3,5-triazine

a. Discussion

(1) In continuation of the study of the reactions of 3,3,3-trinitropropyl amine, the condensation of this amine with formaldehyde

*Aerojet Report No. 660, p. 2.

was investigated. It was found that three moles of 3,3,3-trinitropropyl amine condensed with three moles of formaldehyde to form the cyclic trimer, tris-1,3,5-(3',3',3'-trinitropropyl)-hexahydro-1,3,5-triazine (VIII).



(VIII)

(VIII) is a yellow crystalline compound, mp 121 to 123°C dec., I.S. = 9 cm/2 kg, with a calculated ballistic-mortar value of 140 and a calculated lead-block value of 130.

b. Experimental

(1) In a 300 ml three-necked flask, fitted with a mechanical stirrer and dropping funnel, was placed 34.5 g (0.15 mole) of 3,3,3-trinitropropyl amine hydrochloride, 75 ml of water, and 12.1 g (0.15 mole) of 37% formaldehyde. To this solution was added dropwise a solution of 12.3 g (0.15 mole) of sodium acetate in 50 ml of water. A yellow solid was immediately precipitated, which was collected, washed with water, and dried, to give 30.7 g (99.4%) of product, mp 118 to 123°C dec., I.S . = 9 cm/2 kg. Recrystallization from a large volume of methanol gave yellow plates, mp 121 to 123°C dec.,

Anal. Calc'd for $C_{12}H_{18}N_{12}O_{18}$: %C, 23.31; %H, 2.93; %N, 27.19

Found: %C, 23.78; %H, 2.88; %N, 27.21

Molecular Weight, Calculated: 618

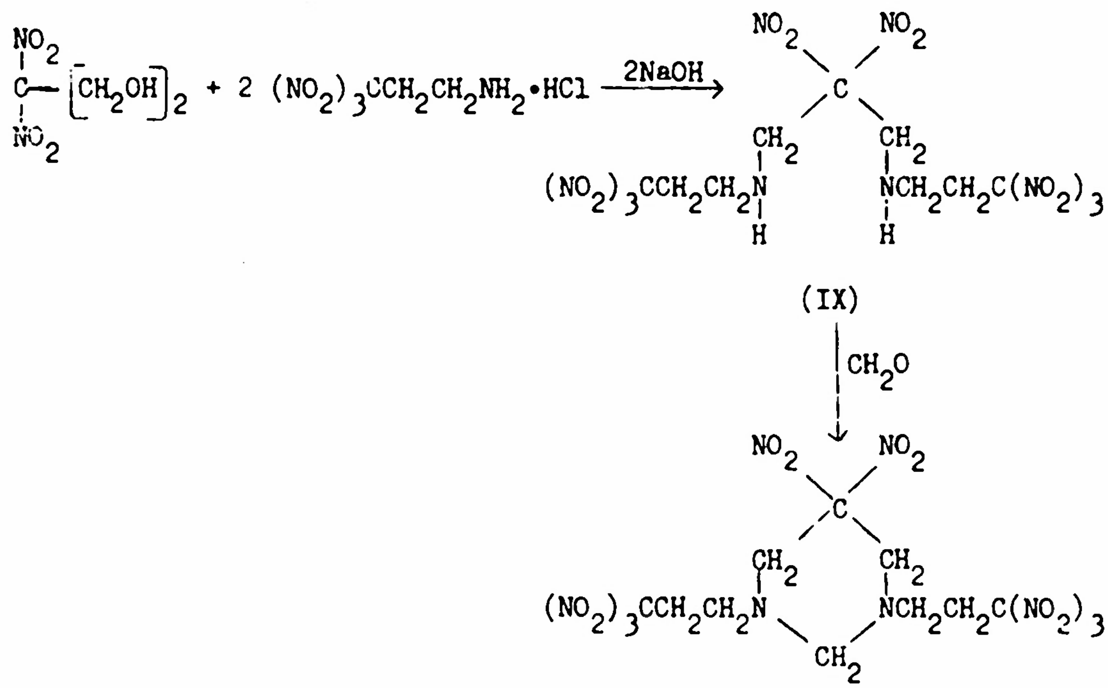
Found: 600, 570

2. Preparation of 1,3-bis(3',3',3'-Trinitropropyl)-5,5-dinitro-hexahydro-1,3-diazine

a. Discussion

(1) It has been shown that the condensation of 3,3,3-trinitropropyl amine with formaldehyde gave 1,3,5-tris(3',3',3'-trinitro-propyl)-hexahydro-1,3,5-triazine (Section II,D,1), while the condensation of

3,3,3-trinitropropyl amine and 2,2-dinitro-1,3-propanediol yielded 1,1,1,6,6,11,11,11-octanitro-4,8-diaza-undecane.* This work prompted a study of the condensation of 1,1,1,6,6,11,11,11-octanitro-4,8-diaza-undecane (IX) with formaldehyde. The product obtained from this condensation, according to its analysis, was postulated to be the desired 1,3-bis(3',3',3'-trinitropropyl)-5,5-dinitro-hexahydro-1,3-diazine (X).



(X)

(X) is a yellow crystalline compound, mp 119 to 121°C dec., I.S. = 5 to 10 cm/2 kg, with a calculated ballistic-mortar value of 157 and a calculated lead-block value of 153.

b. Experimental

(1) In a 300-ml three-necked flask, fitted with a mechanical stirrer, was placed 12.4 g (0.075 mole) of 2,2-dinitro-1,3-propanediol, 34.5 g (0.15 mole) of 3,3,3-trinitropropyl amine hydrochloride, and 75 ml of water. To this solution was added dropwise 30.7 ml of 4.8777N sodium hydroxide solution (0.15 mole). After stirring for 15 minutes a yellow solid had separated. The water was decanted and the product was water-washed by decantation. The solid was dissolved in 200 ml of hot methanol, cooled to 30°C, and 12.1 g (0.15 mole) of 37% formalin was added. The reaction mixture was stirred for one hour; at this time 16.2 g (40.8%) of light-yellow solid had separated,

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*Aerojet Report No. 660, p. 5.

mp 114 to 118°C dec. Evaporation of the methanol solution gave a trace of brown solid. Recrystallization of the product from chloroform gave yellow rods, mp 119 to 121°C dec., I.S. = 5 to 10 cm/2 kg.

Anal. Calc'd for $C_{10}H_{14}N_{10}O_{16}$: %C, 22.65; %H, 2.66; %N, 26.42

Found: %C, 22.95; %H, 2.65; %N, 26.72

E. PREPARATION OF SAMPLES FOR EVALUATION BY THE NAVAL ORDNANCE LABORATORY

1. The following samples were prepared and submitted to the Naval Ordnance Laboratory for evaluation:

a. 1,1,1,4,6,6,8,11,11,11,-Decanitro-4,8-diaza-undecane, 2 g, mp 157 to 159°C dec., I.S. = 5 to 10 cm/2 kg.

b. 1,1,1,3,6,6,9,11,11,11-Decanitro-3,9-diaza-undecane, 7 g, mp 170 to 175°C dec., I.S. = 10 to 15 cm/2 kg.

c. 1,1,1,3,5,7,7,7-Octanitro-3,5-diaza-heptane, 1 g, mp 85 to 86°C, I.S. = 5 to 10 cm/2 kg.

d. 1,1,1,3,6,6,6-Heptanitro-3-aza-hexane, 5.7 g, mp 144 to 145°C, I.S. = 1 cm/2 kg.

e. N-(3',3',3'-Trinitropropyl)-3,3,5,5-tetranitropiperidine, 5 g, mp 144 to 146°C dec., I.S. = 25 cm/2 kg.

f. 3,3,3-Trinitropropyl amine nitrate, 4.3 g, mp 132 to 134°C dec., I.S. = 90 cm/2 kg.

g. 3,3,5,7,7-Pentanitro-5-aza-nonane, 2 g, mp 107 to 108°C, I.S. > 100 cm/2 kg.

2. The conclusions reached by the Naval Ordnance Laboratory with respect to three of the compounds submitted last quarter and six of the compounds submitted this quarter are given below. The data are summarized in Table I and the SPIA data sheets in the appendix.

a. 1,1,1,3,6,6,6-Heptanitro-3-aza-hexane (A52-11)

(1) A52-11 is a primary explosive slightly more sensitive to impact than either mercury fulminate or lead azide. It possesses moderate thermal stability. As a primary explosive it is not initiated readily by a hot wire, which makes it unsuitable for use in electric detonators. The compound is being further evaluated as a primary explosive for stab initiation.

b. 1,1,1,4,6,6-Hexanitro-4-aza-heptane (A52-12)

c. 1,1,1,3,6,6-Hexanitro-3-aza-heptane (A52-13)

A52-12 and A52-13 are isomeric, A52-12 containing the 3,3,3-trinitropropylnitramino grouping and A52-13 the 2,2,2-trinitroethylnitramino grouping. Unfortunately, both compounds melt, with decomposition, slightly above 100°C, and as might be expected, were unstable at 100°C, although A52-13 appeared to decompose at a much faster rate and also to possess a lower ignition temperature than A52-12. The difference in impact sensitiveness is within the range of experimental error and has little significance.

d. N-(3',3',3'-Trinitropropyl)-3,3,5,5-tetranitropiperidine (A52-14)

A52-14 is the third derivative of 3,3,5,5-tetranitropiperidine which has been evaluated. Unfortunately, all the derivatives were very unstable thermally.

e. 3,3,3-Trinitropropyl Amine Nitrate (A52-15)

A52-15 possesses poor thermal stability and is quite sensitive to impact.

f. 3,3,5,7,7-Pentanitro-5-aza-nonane (A52-16)

A52-16 has good thermal stability and is not very sensitive to impact.

g. 1,1,1,4,6,6,8,11,11,11-Decanitro-4,8-diaza-undecane A53-1)

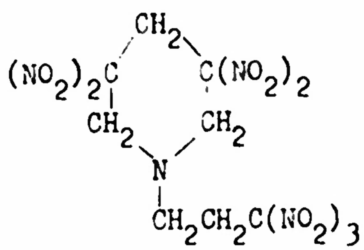
h. 1,1,1,3,6,6,9,11,11,11-Decanitro-3,9-diaza-undecane (A53-2)

i. 1,1,1,3,5,7,7,7-Octanitro-3,5-diaza-heptane (A53-3)

The sensitivities of A53-1, A53-2, and A53-3 are such that their application is questionable. The poor thermal stability of A53-3 now seems to be typical of trinitroethyl nitramines at temperatures above their melting points.

TABLE I

NOL PRELIMINARY TESTS ON HIGH EXPLOSIVES

Compound	mp, °C	I.S. cm/2.5 kg	Cryst. Dens. g cm^{-3}	Ign. Temp °C	Therm. Stab. (Vac., cc/g/48 hr) at 100°C	Calc. Ball.-Mort. Value (RDX=150)	Calc. Pb Block Value (RDX=157)
$(NO_2)_3CCH_2N(NO_2)CH_2CH_2C(NO_2)_3$ (A52-11)	144-145	6.0 (Class 6)	1.829	190	2.98 (Class II)	154*	194*
$(NO_2)_3CCH_2CH_2N(NO_2)CH_2C(NO_2)_2CH_3$ (A52-12)	109-109.5 (dec.)	17.4 (Class 5)	1.652	210	Over 30 in 25 hr (Class IV)	153	162
$CH_3C(NO_2)_2CH_2CH_2N(NO_2)CH_2C(NO_2)_3$ (A52-13)	106-107 (dec.)	19.6 (Class 5)	1.736	173	Over 30 in 2.5 hr (Class IV)	153	162
 (A52-14)	144-146 (dec.)	29 (Class 4)	1.703	—	Over 30 (Class IV)	152	155

$(NO_2)_3CCH_2CH_2NH_2 \cdot HNO_3$ (A52-15)	132-134 (dec.)	10 (Class 6)	1.774	—	Over 30 (Class IV)	144	171
$[CH_3CH_2C(NO_2)_2CH_2]_2NNO_2$ (A52-16)	107-108	80 (Class 3)	1.707	—	1.64 (Class I)	129	112
$(NO_2)_2C[CH_2N(NO_2)CH_2CH_2C(NO_2)_3]_2$ (A53-1)	157-159 (Dec.)	10 (Class 5)	1.81	—	1.87 (Class I)	155	177
$(NO_2)_2C[CH_2CH_2N(NO_2)CH_2C(NO_2)_3]_2$ (A53-2)	170-175 (dec.)	11 (Class 5)	1.84	--	20.6 (Class IV)	155	177
$CH_2[N(NO_2)CH_2C(NO_2)_3]_2$ (A53-3)	84-85	5 (Class 6)	1.87	—	Over 30 in 2 hr (Class IV)	156**	196**

*Adjusted to zero oxygen balance with 5% TNT.

**Adjusted to zero oxygen balance with 12.3% TNT.

III. DESENSITIZATION OF RDX

A. EVALUATION OF SURFACE-ACTIVE AGENTS

1. Discussion

a. A new test of surface activity was employed to evaluate a large number of surface-active agents for possible use in the desensitization of RDX. To determine which agent is best, it is necessary to decide what distinguishes a good surface-active agent for this application. Apparently this question cannot be answered in terms of fundamental physical quantities. As an indirect approach, a test may be selected whose results are believed to correlate with good surface activity, a large number of agents may be subjected to this test, and it may be determined whether the agents known to be good are distinguished by the test.

b. The extent of adsorption on RDX, under conditions comparable to those of actual use, seemed to provide such a test. Previous work in coating with nitropolymers from solution using Span 85, known to be a good surface-active agent, had shown that several percent of Span 85 (on basis of RDX) was needed to produce optimum results, and that most of the Span was adsorbed on the RDX, with only a small amount left in the liquid phase.

c. Preliminary experiments in a medium of ethanol and water have been described in Report No. 660. They showed that Span 85 gave a relatively high adsorption (25% average) in comparison with, for example, Tween 20 (6%). However, it was desirable to enlarge the spread of values between good and bad agents, in order to obtain greater distinction.

d. In the work of the current quarter, the change was made to a medium of acetone and water. In the new medium, values of the adsorption of Span 85, while not satisfactorily reproducible, were larger on the average than the amounts adsorbed from ethanol and water. However, for some other agents the values of adsorption were consistently reproducible. For a broader view of the usefulness of the test, it was applied, in this new form, using the acetone-water medium, to a group of 37 other surface-active agents. Most of these were experimental materials of various chemical types provided by the Atlas Powder Company. The percent of each agent adsorbed on RDX from the acetone-water medium was measured, as was the impact stability of RDX plus the adherent agent.

e. To show the effect of concentration of surface-active agent on its distribution between liquid and solid in this test, other trials were made with about 1/2 and 1/5 the amounts of agent used in the standard procedure. Unfortunately, the agents tested were Span 85 and one other of similar behavior, and poor reproducibility obscured the meaning of the results.

f. A comparison was also made between the values of percent adsorbed and the values of "hydrophile-lipophile balance" (HLB) for all agents from the Atlas Powder Company for which these figures are available.

g. In a separate experiment the amounts adsorbed on RDX from a water-acetone-ethyl acetate medium of Span 85 alone were compared with the amounts adsorbed in the presence of precipitating polyurethane I-A. The amounts were similar, and possibly identical within experimental error.

2. Conclusions

a. Consideration of the results of the standard adsorption test with acetone-water for the whole body of agents showed them to fall into fairly distinct groups, distinguished as follows:

Type	Behavior in Medium	% Adsorbed*	Impact Stability** cm/2 kg
(1) Several types	Solution	3-16	20-50
(2) Several types	Suspension	4-30	30-45
(3) Esters	Suspension	16-91	35- >100
(4) Esters	Suspension + separate oil phase	85-99	>100

*Recovery of original agent, which is 5% of RDX.

**30 cm/2 kg for RDX.

b. Probably the best agents lie in Group (3), which includes Span 85 and Prosol 307, among others. It is believed that the separation of a distinct oil layer noted in agents of Group (4) shows too high a lipophilic tendency and is a disadvantage.

c. Trials showed that the reproducibility of the test, using three agents from Group (3), was very poor (16 to 73% adsorbed for Span 85). Trials with single agents from each of the other groups showed the reproducibility to be good.

d. The poor reproducibility of the test for compounds in Group (3) might be considered to result from a delicate balance between the tendency to remain suspended and the tendency to adhere to RDX, with a small influence sufficing to swing the balance either way. In Groups (1) and (2) the former tendency predominates, and in Group (4) the latter.

e. Considering the relation of impact stability to percent adsorbed, there is a narrow margin between negligible and sizable desensitization within the adsorption range covered by agents of Group (3). This transition occurs at an adsorption of about 40% in the standard procedure (i.e., for adsorption of about 2% of the RDX, on an absolute basis). This relation appears to be independent of the chemical or physical nature of the agents.

f. These data emphasize the fact that in work to date on coating RDX with nitropolymers, a large share of the desensitization has been provided by the surface-active agent. The need for new and better nitro-compound coatings is **emphasized.**

g. The percentage adsorbed correlates roughly with the Atlas hydrophile-lipophile balance. All compounds for which the standard procedure gives adsorptions of 15% or greater have low HLB values, i.e., less than 6. All evidence to date indicates that these are the most useful compounds. (On the other hand, two compounds show low HLB values combined with low percentages adsorbed.)

3. Experimental

a. The adsorption of 0.15 g of surface-active agent on 3.00 g RDX from a medium of 5 ml acetone plus 10 ml water was determined, for 38 surface-active agents, by the method described in detail in Report No. 660. From each experiment, portions of RDX plus adherent agent were set aside, dried, and tested for impact stability with the Bureau of Mines apparatus. Allowance was made for the removal of these portions in the calculation of percents adsorbed. Results of these experiments are summarized in Table II.

b. For several of the agents - Span 85, G-949, Prosol-307, G-1226, G-2266, and G-1071 - the experiment was repeated once or more to test the reproducibility of the method. Results of these replicate experiments are also summarized in the table.

c. The effect on adsorption of diminishing the amount of surface-active agent from 5% of the RDX (in the standard procedure of a, above) to 2-1/2 and 1% was investigated. A few results obtained with Span 85 and G-949 are given in Table III.

d. Values of the hydrophile-lipophile balance (HLB) for many of the agents listed in Table II were provided by the Atlas Powder Company. Table IV lists these HLB values, comparing them with percentages adsorbed from Table II.

e. In a separate experiment, 0.083 g of Span 85 was dissolved in 10 ml acetone in a 100 ml flask. To this was added 30 ml water, and 2.00 g RDX was suspended in the mixture. It was stirred for 5 minutes, and 0.10 g polyurethane I-A dissolved in 4 ml ethyl acetate was added slowly over an interval of 15 minutes. The solid phase was separated and dried, and the adsorbed Span 85 was recovered by the usual extraction procedures. The amount adsorbed was 86%. In a parallel experiment, similar in every respect except that ethyl acetate alone, with no dissolved polymer, was added, the amount adsorbed was 73%.

TABLE II

ADSORPTION OF SURFACE-ACTIVE AGENTS ON RDX

(Adsorption of 0.15 g agent on 3.00 g RDX from a medium of 5 ml acetone + 10 ml water)

Run	Agent	Type	Physical Form	Appearance of Agent in Medium	% Adsorbed	I.S.* cm/2 kg
11/7/1	Atlas G-3910	Polyoxyethylene fatty alcohol	Fluid suspension	Solution	5	20-25
11/7/2	Atlas G-3915	Polyoxyethylene fatty alcohol	Fluid suspension	Solution	3	40
11/7/3	Atlas G-3920	Polyoxyethylene fatty alcohol	Mush	Solution	7	25
11/12/1	Brij 30	Polyoxyethylene fatty alcohol	Liquid	Solution	7	35
11/13/1	Brij 35	Polyoxyethylene fatty alcohol	Wax	Solution	6	35-40
11/14/1	Atlas G-1690	Polyoxyethylene alkyl aryl ether	Liquid	Solution	8	35-40
11/6/2	Atlas G-1065	Polyoxyethylene sorbitol ester	Liquid	Solution	6	30-35 **
11/14/2	Atlas G-1071	Polyoxyethylene sorbitol ester	Liquid	Suspension + oil	90	>100 (30)
1/2/1					91	—
1/2/2					85	--
11/17/1	Atlas G-1077	Polyoxyethylene sorbitol ester	Liquid	Suspension	14	35-45
11/17/2	Atlas G-1083	Polyoxyethylene sorbitol ester	Liquid	Solution	7	30-35
11/17/3	Atlas G-1085	Polyoxyethylene sorbitol ester	Liquid	Solution or suspension	9	30
11/17/4	Atlas G-1086	Polyoxyethylene sorbitol ester	Liquid	Solution or suspension	9	35-40
11/18/1	Atlas G-1087	Polyoxyethylene sorbitol ester	Liquid	Suspension	6	30-35
11/18/2	Atlas G-1095	Polyoxyethylene sorbitol ester	Liquid	Solution	8	30-35
11/21/1	Atlas G-1096	Polyoxyethylene sorbitol ester	Liquid	Solution	11	30-50
11/21/2	Atlas G-2139	Polyoxyethylene fatty acid	Liquid	Suspension	30	40-45
11/24/1	Atlas G-2140	Polyoxyethylene fatty acid	Liquid	Suspension	12	35-40
11/24/2	Atlas G 2142	Polyoxyethylene fatty acid	Liquid	Solution	3	35-40
11/24/3	Atlas G-2144	Polyoxyethylene fatty acid	Mush	Solution	5	30-35
11/24/4	Atlas G-2266	Polyoxyethylene fatty-acid ester	Mush	Suspension	9	30-35
1/2/3					6	—
1/2/4					3	—
11/24/5	Atlas G-2269	Polyoxyethylene fatty-acid ester	Wax	Solution	5	40
11/24/6	Atlas G-1216	Polyoxyethylene tall oil	Liquid	Suspension	6	35
11/25/1	Atlas G-1226	Polyoxyethylene tall oil	Liquid	Solution	5	30-45
1/5/1					4	—
1/5/2					5	—
11/25/2	Atlas G-1234	Polyoxyethylene recinoleate	Liquid	Solution	4	40-45
11/25/3	Atlas G-1286	Polyoxyethylene recinoleate	Wax	Solution	4	30-35
11/25/4	Atlas G-271	Cationic	Liquid	Solution or suspension	6	40-45
11/26/1	Atlas G-263	Cationic	Liquid (+ slight precipitate)	Solution	4	35
11/26/2	Atlas G-650	Sorbitan ester	Liquid-mush	Suspension	6	40
11/5/2	Atlas G-949	Sorbitan ester	Liquid	Suspension	73	>100
12/19/4					37	35-40
12/23/2					43	—
11/26/3	Atlas G-992	Sorbitan ester	Liquid	Suspension	4	30-35
11/26/4	Arlacel C	Sorbitan ester	Liquid	Suspension	8	30-35
11/28/1	NNO	Sorbitan ester	Liquid	Suspension	11	30-35 **
11/3/1	Span 85	Sorbitan ester	Liquid	Suspension	61	100 (25)
12/18/1					16	—
12/19/2					21	—
12/22/2					63	100
12/22/3					37	35-50
12/22/4					17	45-50
11/28/2	Atlas G-3865	Polyoxyethylene fatty amine	Liquid	Solution	5	30-45
11/5/3	Atlas G-3785	Polyoxyethylene fatty amide	Liquid-mush	Solution	16	35-40
11/28/3	Atlas G-3300	Alkyl aryl sulfonate	Liquid	Solution	3	35-40
11/5/1	Prosol 307	Polyglycol ester	Liquid	Suspension	72	>100
					91	>100
11/6/1	Prosol DO 106	Polyglycol ester	Liquid	Suspension + oil	99	>100

*For RDX, 30 cm/2 kg.

**On 5/0 sandpaper.

TABLE III

EFFECT OF DECREASING THE AMOUNTS OF SURFACE-ACTIVE AGENTS ON THEIR ADSORPTION ON RDX

(Adsorption on 3.00 g RDX from a medium of 5 ml acetone + 10 ml water)

Run	Agent	Type	% Agent (RDX Basis)	Appearance of Agent in Medium	% Adsorbed
12/18/2	Span 85	Sorbitan ester	2.5	Suspension	10
12/23/1					14
12/18/3	Span 85	Sorbitan ester	ca. 1	Suspension	7
12/19/1	G-949	Sorbitan ester	2.5	Suspension	33

TABLE IV

HYDROPHILE-LIPOPHILE BALANCES OF SOME SURFACE-ACTIVE AGENTS, AND THEIR RELATION TO AMOUNTS ADSORBED ON RDX (FROM TABLE I)

Agent	HLB	% Adsorbed	Agent	HLB	% Adsorbed
G-3910	12.2	5	G-2142	11.1	3
G-3915	14.1	3	G-2144	15.0	5
G-3920	15.2	7	G-1216	8.4	6
G-1065	8.5	6	G-1226	13.5	5
G-1071	5.3	85-91	G-1284	13.5	4
G-1077	8.0	14	G-1286	14.0	4
G-1083	13.8	7	G-263	35-40	4
G-1085	11.3	9	G-650	2.5	6
G-1086	10.2	9	G-949	3.7	37-73
G-1087	9.2	6	G-992	2.7	4
G-1095	12.4	8	—	—	--
G-1096	11.4	11	Arlacel C	3.7	8
G-2139	5.1	30	Span 85	1.8	16-63
G-2140	8.0	12	G-3865	13.0	5
--	—	—	G-3300	11.7	3

B. THE COATING OF RDX

1. Discussion

a. Two samples of desensitized RDX, S-234 and S-235, prepared last quarter,* have been evaluated by the Naval Ordnance Laboratory. The RDX was densitized using a 10% coating, applied from solution at 85°C, of a co-polymer containing 90% 2,2-dinitrobutyl acrylate and 10% 2-nitrobutyl acrylate with 2.5% Span 85 as a wetting agent. The impact sensitivities of A-234 and S-235 were found to be 56 and 43 centimeters, respectively. The sensitivity of Composition A by this procedure, in which a 2.5-kilogram weight was used, was 59 centimeters. The detonation rate vs density curve for e ach of these samples will be determined by NOL.

b. A new batch of RDX, Wabash Lot No. 119, has been received. The desensitization was carried out in the same manner as was used on S-234 and S-235, but the results were not as good. The new RDX coated with the co-polymer gave a vlue of 25 to 30 cm/2 kg on 5/o sandpaper, as compared with the previous values of 30 to 50 cm/2 kg. This may be attributed to the fact that the average particle size of the new batch of RDX is smaller than that of the old.

*Aerojet Report No. 660, p. 36.

c. Six new compounds, 3,3,7,7-tetranitro-5-aza-nonane, 3,3,5,7,7-pentanitro-5-aza-nonane, 4,4,8,8-tetranitro-6-aza-undecane, 4,4,6,8,8-pentanitro-6-aza-undecane, dimethyl 4,4,12,12-tetranitro-7,9-diaza-8-keto-pentadecanedioate, and 5,5-dinitrovalerolactone were tried as coating agents for RDX, using Span 85 as a wetting agent. Some desensitization was achieved, the best result being obtained with 3,3,5,7,7-pentanitro-5-aza-nonane, which gave an impact stability of 30 to 35 cm/2 kg on 5/o sandpaper.

d. In the evaluation of new surface-active agents (III,A) some promising new wetting agents were discovered. Four of these Atlas wetting agents were tested in the coating of RDX with poly 2,2-dinitrobutyl acrylate. The impact stabilities of the coated samples ranged from 60 to 100 cm/2 kg, which was an improvement over the runs in which Span 85 (I.S. = 60 to 65 cm/2 kg) was used. These new wetting agents will be evaluated further, particularly in the case of the co-polymer of 2,2-dinitrobutyl acrylate and 2-nitrobutyl acrylate.

2. Experimental

a. In a 500-ml resin pot, fitted with a mechanical stirrer, dropping funnel, and thermometer, was placed 50 ml of water. Stirring was started, the water was heated to 85°C on the steam bath, and the wetting agent was added. After two minutes the RDX was added. In five minutes a solution of the coating agent in 25 ml of methyl isobutyl ketone (acetone was used as the solvent in S-256 and S-257) was added dropwise. After all the solvent had been removed by azeotropic distillation, the coated RDX was collected, washed with water, and dried. The experimental results are summarized in Table V.

IV. DEVELOPMENT OF BOMB AND SHELL CASINGS MADE OF AN EXPLOSIVE PLASTIC

The third objective of the explosives program is the development of bomb and shell casings made of an explosive plastic. Thus, if a nitropolymer were used for the casing instead of metal, the explosive plastic would contribute toward the force of the explosion rather than being merely dead weight. Up to the present, this phase of the program has received little priority, since it is dependent on the progress made on the synthesis of nitropolymers which have physical and mechanical properties suitable for use in a bomb or shell casing. Until the middle of last year, the only nitropolymers prepared were too low in molecular weight to possess enough physical and mechanical strength for use as explosive plastics. Recent work on the nitropolymer program has shown that the use of chelated metals as catalysts in polyurethane formation has made it possible to obtain nitropolymers with a molecular weight of 50,000 which have considerably improved physical and mechanical properties.* At this point it is possible to initiate some work on Phase Three of the explosives program, and in the future the development of explosive plastics will be reported.

*Aerojet Report No. 663, p. 13.

TABLE V

DESENSITIZATION OF RDX

Sample No.	Coating Agent	% Coating	Wetting Agent	% Wetting Agent	Temp °C	Water/RDX Ratio	I.S., cm/2 kg Regular	I.S., cm/2 kg 5/0 Sandpaper
S-240	1	10	Span 85	2.5	85	2.5/1	55-60	--
S-241	2	10	Span 85	2.5	85	2.5/1	100	30-35
S-242	90% DNBA[3]							
	10% MNBA[4]	10	Span 85	0.5	85	2.5/1	100	25
S-243[5]	DNBA	10	Span 85	2.5	85	2.5/1	60-65	—
S-246	DNBA	10	Span 85	2.5	85	2.5/1	60-65	—
S-244	90% DNBA							
	10% MNBA	10	Span 85	2.5	85	2.5/1	>100	20-25
S-247	10% MNBA	10	Span 85	2.5	85	2.5/1	>100	25-30
S-245	80% DNBA							
	20% MNBA	10	Span 85	2.5	85	2.5/1	>100	25-30
S-248	20% MNBA	10	Span 85	2.5	85	2.5/1	>100	25-30
S-249	20% MNBA	10	Span 85	2.5	85	12.5/1	>100	25-30
S-250	6	10	Span 85	2.5	85	2.5/1	>100	25-30
S-251	7	10	Span 85	2.5	85	2.5/1	>100	25-30
S-252	DNBA	10	Atlas G-1077	2.5	85	2.5/1	75-80	—
S-253	DNBA	10	Atlas G-2139	2.5	85	2.5/1	75-80	--
S-254	DNBA	10	Atlas G-949	2.5	85	2.5/1	100	—
S-255	DNBA	10	Atlas G-3785	2.5	85	2.5/1	85-90	--
S-256	8	10	Span 85	2.5	85	2.5/1	60-65	—
S-257	9	10	Span 85	2.5	85	2.5/1	90-95	—

[1] 3,3,7,7-Tetranitro-5-aza-nonane.

[2] 3,3,5,7,7-Pentanitro-5-aza-nonane.

[3] 2,2-Dinitrobutyl acrylate.

[4] 2-Nitrobutyl acrylate.

[5] Starting with S-243 a new batch of RDX, Wabash Lot No. 119, was used.

[6] 4,4,8,8-Tetranitro-6-aza-undecane

[7] 4,4,6,8,8-Pentanitro-6-aza-undecane.

[8] Dimethyl 4,4,12,12-tetranitro-7,9-diaza-8-keto-pentadecanedioate (Aerojet Report in press).

[9] 5,5-Dinitrovalerolactone, Klager, J. Org. Chem. 16, 161 (1951).

N7onr-462, Task Order I

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Standard Oil Company Research Department P. O. Box 431 Whiting Indiana Attn: Dr. W. H. Bahlke	1
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Dr. T. L. Brownyard Bureau of Ordnance, Re2c Department of the Navy Washington 25, D. C.	1
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Dr. N. L. Drake Department of Chemistry University of Maryland College Park, Maryland	1
Dr. A. V. Tobolsky Frick Chemical Laboratory Princeton University Princeton, New Jersey	1

Report No. 682
SPIA/M3

Data Questionnaire on

COMPOUNDS FOR USE AS INGREDIENTS OF PROPELLANTS AND OTHER EXPLOSIVES

Listed below are the characteristics which are believed to be of importance in the study of a compound which may possibly be used as a constituent of solid propellants or other explosives. It is not expected that all this information will be available for every compound. For example, with a compound showing poor stability, it is probably not advisable to investigate that compound farther. After placing such informati as is available for a compound which you have prepared or tested on one of these forms, send it to SOLID PROPELLANT INFORMATION AGENCY, APL/JHU, 8621 Georgia Avenue, Silver Spring, Maryland. As additional information on the same or new compounds accrues, forward it on a similar form at a later date. The information submitted on these forms will be rewritten and published by SPIA in loose-leaf manual form. These forms may also be used as work or data sheets for your experimental studies. Extra copies are available upon request from SPIA. Suggestions for improvement of these forms are invited. If insufficient space has been provided for any item, attach separate sheets.

COMPOUND:

Name 1,1,1,3,6,6,6-Heptanitro-3-aza-hexane

Empirical formula $C_5H_6N_8O_{14}$

Structure: (configuration)

$$(NO_2)_3CCH_2CH_2\overset{\displaystyle NO_2}{\overset{|}{N}}CH_2C(NO_2)_3$$

Information submitted by:
Activity Aerojet Engineering Corporation
Person M.B. Frankel and L.T. Carleton
Date 10 December, 1952

Preparation reaction(s):

$$(NO_2)_3CCH_2CH_2NH_2\cdot HCl + HOCH_2C(NO_2)_3 \xrightarrow{NaOH} (NO_2)_3CCH_2CH_2\overset{\displaystyle H}{\overset{|}{N}}CH_2C(NO_2)_3 \xrightarrow{HNO_3} (NO_2)_3CCH_2CH_2\overset{\displaystyle NO_2}{\overset{|}{N}}CH_2C(NO_2)_3$$

1. Quantitative analysis: (% by weight)

	Carbon	Hydrogen	Oxygen	Nitrogen		
Calculated from formula	14.93	1.50	55.71	27.86		
By determination	15.23	1.60		28.13		

2. Burning properties: (compared to nitrocellulose, under nitrogen at atmospheric pressure:)

(faster? slower? residue? etc.)

3. Stability and Sensitivity: Plot any graphs on separate sheet. Discuss methods used when they vary from references. Give temperature used. (Use separate sheet if necessary.)

	Name of test	Recommended method	
a.	Impact Sensitivity	NOL [illegible]	2.5-kg weight
b.	Thermal Stability	OSRD 3401 p.8	
c.	Vacuum Stability	NOL [illegible]	48 hr at 100°C
d.	Temperature of Explosion	OSRD 3401 p.6	
e.	Temperature of Ignition	NOL [illegible]	
f.			
g.	Impact Stability	Bureau of Mines Bull.	No. 346, p. 72; 2-kg weight
h.			

RESULTS OF ABOVE TESTS

	Reference compound (designation-TNT, Tetryl, N.C., etc.)	New Compound test results
a.	Tetryl, 32 cm/2.5 kg	6.0 cm/2.5 kg
b.		
c.		2.98 cc/g
d.		
e.		190°C
f.		
g.	RDX, 30 cm/2 kg	1-2 cm
h.		

4. Heat of formation: (ΔH) ± −28.1 (Indicate sign) Kg. calories at 25°C., 1 atm. pressure

Report No. 682
SPIA/M3

		By Experiment		By Calculation	Method (Description or reference. Separate sheet if necessary.)
5.	Energy of explosion (Q) (at 25°C, H_2O liquid)		cal/gm		
6.	Heat of combustion (H_c) (at 25°C, H_2O liquid)	1609	cal/gm	1611	Aerojet Report No. 417A
7.	Specific impulse (I_{sp}) calc:		lb-sec/lb		

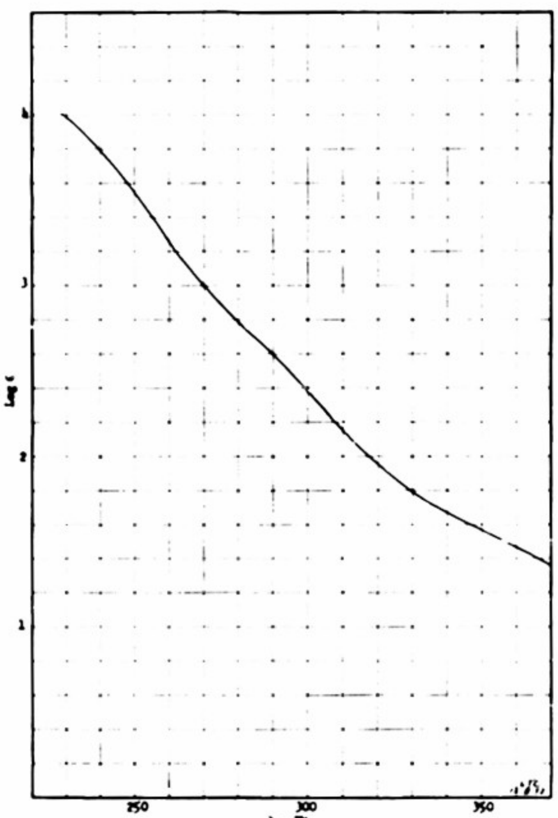
8. Physical form of compound (viscous liquid, crystalline type, etc.) White needle-shaped crystals

9. Simple microscope analysis data: (crystal studies)

10. Density (Macro method) 1.83 gm/cm³. (Micro or other method) ________ gm/cm³. (Explain on separate sheet any unique methods you use.)

11. Index of refraction: ($n_D^{25°C.}$) ________ 12. Color ________ 13. Odor None.

14. pH at 25°C. 0.8 (Method reference OSRD 3401 p.4, or OSRD 5968. Indicate method used, i.e. solvent and concentrations used, pH indicator paper or Beckman pH meter.) Beckman pH meter; 0.0077M in acetone/water (5/1 volume ratio)



Ultraviolet Absorption Spectrum of 1,1,1,3,6,6,6-Heptanitro-3-aza-hexane in Ethanol

17. Boiling point, or decomposition temperature: ________ °C. (underline which temperature is reported)

18. Heat of Vaporization: ________ g-cal./gm ________ BTU/lb.

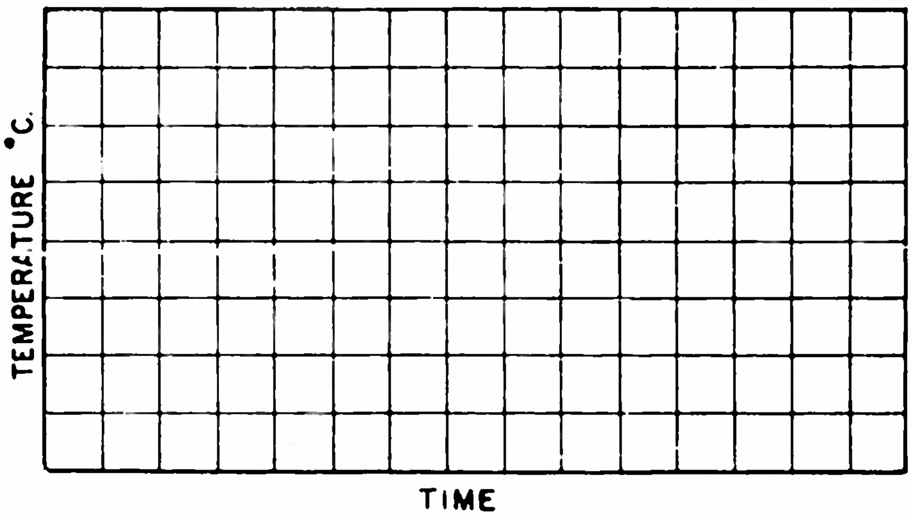
19. Heat of Fusion: ________ g-cal./gm ________ BTU/lb.

20. Melting point 144-145 °C.

Page 2
Appendix

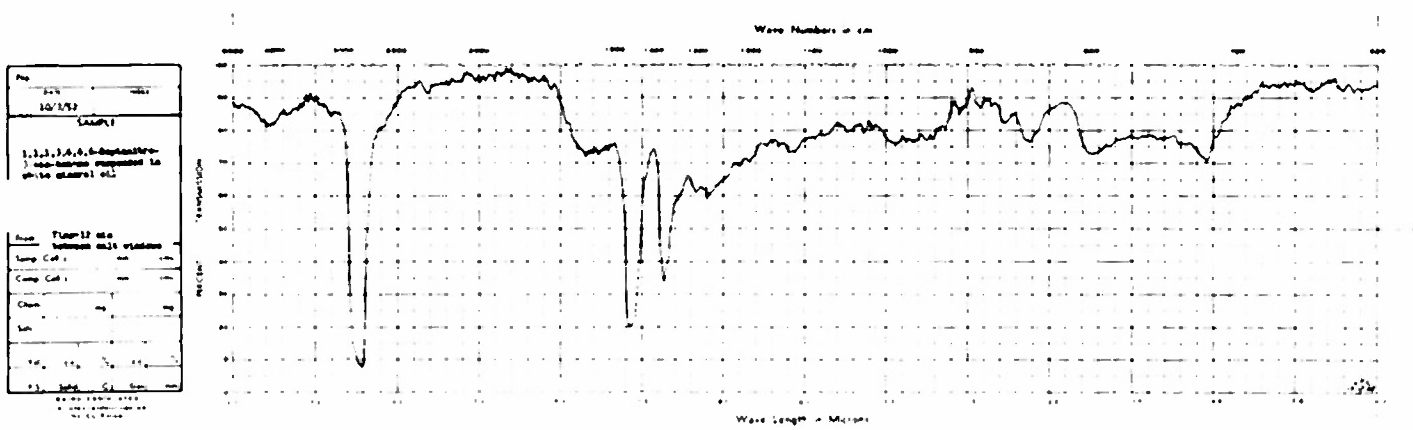
Report No. 682
SPIA/M3

21. Freezing point vs. Time Curve: (Please mark the coordinates with scale values that apply to the compound under study.)



22. Solubility of new compound:

<0.1	g/100 ml H_2O at 25°C.			g/100 ml H_2O at		°C.
120	g/100 ml	acetone (name material used as solvent)	at		°C.	
0.6	g/100 ml	toluene (name material used as solvent)	at		°C.	



1,1,1,3,6,6,6-Heptanitro-3-aza-hexane Suspended in White Mineral Oil
Time=12 min

Under compatability we are considering the ability of two compounds to be in intimate contact (Note OSRD 5758 p. 21-22) over a long period of time without adverse effects on either the chemical or physical properties of either material. These tests will probably be of varied extent. They might be some of the following: (a) Standard stability tests. (b) Simple observations of exudation or separation at ambient or accelerated temperature. (c) Prepare thin sheets (.025" thick, 1" square) of the plasticized material. Separate the sheets with strips of cigarette paper or carbon paper and compress the stack in a "C" clamp. After several days note the oily collection on the paper. Please give reference to or describe procedure used. Item 28 is to indicate results when the compound is in contact with some material other than ethyl cellulose, nitrocellulose or rubber.

25. Compatability with ethyl cellulose: ______

26. Compatability with nitrocellulose: ______

27. Compatability with rubber: ______

28. Compatability with ______: ______

29. Polymerizing properties of the new compound:
   (a) By itself ______
   (b) In mixtures (with additives) ______
   (c) Inhibiting action on polymerization of:
      Thiokol ______
      Methacrylate ______
      Other compounds ______

30. Availability
   a. Amount now available? Research quantities
   b. When was available material first prepared? ______
   c. Amount prepared at that time? ______
   d. Is large production feasible? ______
   e. Plant capacity in existance, lbs/day? ______
   f. Outline steps for a quantity production method ______

31. Additional information: (toxicity, hazards, deterioration, oxygen balance, detonation rate, explosive power, ease of hydrolysis by water, etc. List references, reports, data books, etc. that refer to the compound.)

Oxygen balance = +4
When adjusted to zero oxygen balance with 5% TNT, the calculated lead-block value is 194 and the calculated ballistic-mortar value is 154 (method of Aerojet Report No. 512, p. 8).

Data Questionnaire on

COMPOUNDS FOR USE AS INGREDIENTS OF PROPELLANTS AND OTHER EXPLOSIVES

Listed below are the characteristics which are believed to be of importance in the study of a compound which may possibly be used as a constituent of solid propellants or other explosives. It is not expected that all this information will be available for every compound. For example, with a compound showing poor stability, it is probably not advisable to investigate that compound farther. After placing such information as is available for a compound which you have prepared or tested on one of these forms, send it to SOLID PROPELLANT INFORMATION AGENCY, APL/JHU, 8621 Georgia Avenue, Silver Spring, Maryland. As additional information on the same or new compounds accrues, forward it on a similar form at a later date. The information submitted on these forms will be rewritten and published by SPIA in loose-leaf manual form. These forms may also be used as work or data sheets for your experimental studies. Extra copies are available upon request from SPIA. Suggestions for improvement of these forms are invited. If insufficient space has been provided for any item, attach separate sheets.

COMPOUND:

Name 1,1,1,4,6,6-Hexanitro-4-aza-heptane

Empirical formula $C_6H_9N_7O_{12}$

Structure: (configuration)

$(NO_2)_3CCH_2CH_2N(NO_2)CH_2C(NO_2)_2CH_3$

Information submitted by:

Activity Aerojet Engineering Corporation

Person M.B. Frankel and L.T. Carleton

Date 10 December, 1952

Preparation reaction(s):

$(NO_2)_3CCH_2CH_2NH_2\cdot HCl + HOCH_2C(NO_2)_2CH_3 \xrightarrow{NaOH} (NO_2)_3CCH_2CH_2NHCH_2C(NO_2)_2CH_3 \xrightarrow{HNO_3} (NO_2)_3CCH_2CH_2N(NO_2)CH_2C(NO_2)_2CH_3$

1. Quantitative analysis: (% by weight)

	Carbon	Hydrogen	Oxygen	Nitrogen		
Calculated from formula	19.41	2.44	51.73	26.42		
By determination	19.90	2.52		26.91		

2. Burning properties: (compared to nitrocellulose, under nitrogen at atmospheric pressure:)

(faster? slower? residue? etc.)

3. Stability and Sensitivity: Plot any graphs on separate sheet. Discuss methods used when they vary from references. Give temperature used. (Use separate sheet if necessary.)

	Name of test	Recommended method	
a.	Impact Sensitivity	NOL ~~OSRD [illegible]~~	2.5-kg weight
b.	Thermal Stability	OSRD 3401 p.8	
c.	Vacuum Stability	NOL ~~OSRD 3401 p.10~~	48 hr (or less) at 100°C
d.	Temperature of Explosion	OSRD 3401 p.6	
e.	Temperature of Ignition	NOL ~~OSRD 3401 p.6~~	
f.			
g.	Impact Stability	Bureau of Mines Bull. No. 346, p. 72; 2-kg weight	
h.			

RESULTS OF ABOVE TESTS

	Reference compound (designation-TNT, Tetryl, N.C., etc.)	New Compound test results
a.	Tetryl, 32 cm/2.5 kg	17.4 cm/2.5 kg
b.		
c.		>30 cc/g in 25 hr
d.		
e.		210°C
f.		
g.	RDX, 30 cm/2 kg	35-40 cm/2 kg
h.		

4. Heat of formation: (ΔH) ± -18.7 (indicate sign) Kg. calories at 25°C., 1 atm. pressure

Report No. 682
SPIA/M3

	By Experiment		By Calculation	Method (Description or reference. Separate sheet if necessary.)
5. Energy of explosion (Q) (at 25°C, H_2O liquid)		cal/gm		
6. Heat of combustion (H_c) (at 25°C, H_2O liquid)	2298	cal/gm	2232	Aerojet Report No. 417A
7. Specific impulse (I_{sp}) calc:		lb-sec/lb		

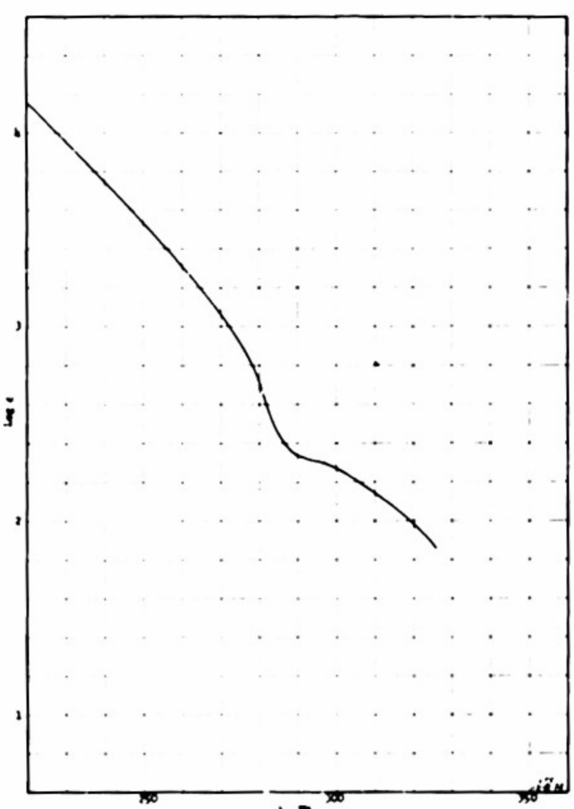
8. Physical form of compound (viscous liquid, crystalline type, etc.) Crystalline in faintly orange plates.

9. Simple microscope analysis data: (crystal studies)

10. Density (Macro method) 1.65 gm/cm³. (Micro or other method) ______ gm/cm³. (Explain on separate sheet any unique methods you use.)

11. Index of refraction: ($n_D^{25°C.}$) ______ 12. Color ______ 13. Odor None.

14. pH at 25°C. 4.4 (Method reference OSRD 3401 p.4, or OSRD 5968. Indicate method used, i.e. solvent and concentrations used. pH indicator paper or Beckman pH meter.) Beckman pH meter; 0.0014M in acetone/water (5/1 volume ratio)



Ultraviolet Absorption Spectrum of 1,1,1,[illegible] in Ethanol

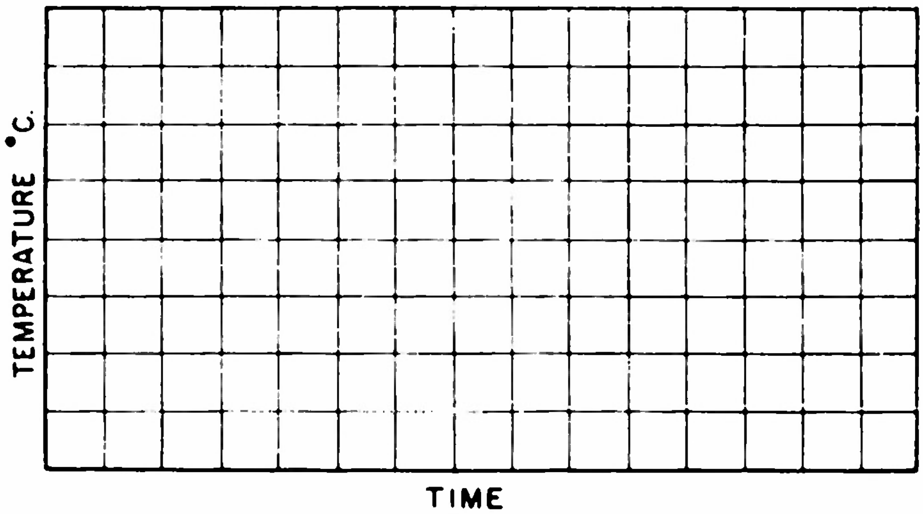
17. Boiling point, or decomposition temperature: Decomposes on melting °C. (underline which temperature is reported)

18. Heat of Vaporization: ______ g-cal./gm ______ BTU/lb.

19. Heat of Fusion: ______ g-cal./gm ______ BTU/lb.

20. Melting point: 109-109.5 °C.

21. Freezing point vs. Time Curve: (Please mark the coordinates with scale values that apply to the compound under study.)



22. Solubility of new compound:

<0.1 g/100 ml H_2O at 25°C. ______ g/100 ml H_2O at ______ °C.

200 g/100 ml acetone at 25 °C.
(name material used as solvent)

4.0 g/100 ml toluene at 25 °C.
(name material used as solvent)

1,1,1,4,6,6-Hexanitro-4-aza-heptane Suspended in White Mineral Oil
Time=12 min

Under compatability we are considering the ability of two compounds to be in intimate contact (Note OSRD 5758 p. 21-22) over a long period of time without adverse effects on either the chemical or physical properties of either material. These tests will probably be of varied extent. They might be some of the following: (a) Standard stability tests. (b) Simple observations of exudation or separation at ambient or accelerated temperature. (c) Prepare thin sheets (.025" thick, 1" square) of the plasticized material. Separate the sheets with strips of cigarette paper or carbon paper and compress the stack in a "C" clamp. After several days note the oily collection on the paper. Please give reference to or describe procedure used. Item 28 is to indicate results when the compound is in contact with some material other than ethyl cellulose, nitrocellulose or rubber.

25. Compatability with ethyl cellulose: ______

26. Compatability with nitrocellulose: ______

27. Compatability with rubber: ______

28. Compatability with ______: ______

29. Polymerizing properties of the new compound:
   (a) By itself ______
   (b) In mixtures (with additives) ______
   (c) Inhibiting action on polymerization of:
      Thiokol ______
      Methacrylate ______
      Other compounds ______

30. Availability
   a. Amount now available? Research quantities
   b. When was available material first prepared? ______
   c. Amount prepared at that time? ______
   d. Is large production feasible? ______
   e. Plant capacity in existance, lbs/day? ______
   f. Outline steps for a quantity production method ______

31. Additional information: (toxicity, hazards, deterioration, oxygen balance, detonation rate, explosive power, ease of hydrolysis by water, etc. List references, reports, data books, etc. that refer to the compound.)

Calculated Lead-Block Value = 162
Calculated Ballistic-Mortar Value = 153
Method of Aerojet Report No. 512, p. 8.

Report No. 682
SPIA/M3

Data Questionnaire on

COMPOUNDS FOR USE AS INGREDIENTS OF PROPELLANTS AND OTHER EXPLOSIVES

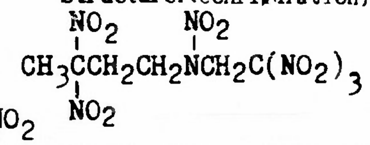
Listed below are the characteristics which are believed to be of importance in the study of a compound which may possibly be used as a constituent of solid propellants or other explosives. It is not expected that all this information will be available for every compound. For example, with a compound showing poor stability, it is probably not advisable to investigate that compound farther. After placing such information as is available for a compound which you have prepared or tested on one of these forms, send it to SOLID PROPELLANT INFORMATION AGENCY, APL/JHU, 8621 Georgia Avenue, Silver Spring, Maryland. As additional information on the same or new compounds accrues, forward it on a similar form at a later date. The information submitted on these forms will be rewritten and published by SPIA in loose-leaf manual form. These forms may also be used as work or data sheets for your experimental studies. Extra copies are available upon request from SPIA. Suggestions for improvement of these forms are invited. If insufficient space has been provided for any item, attach separate sheets.

COMPOUND:

Name 1,1,1,3,6,6-Hexanitro-3-aza-heptane

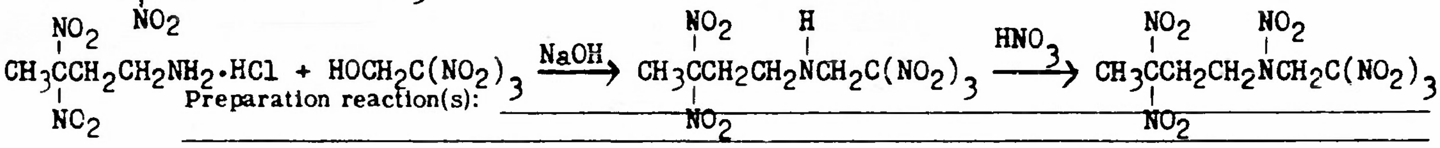
Empirical formula $C_6H_9N_7O_{12}$

Structure: (configuration)



Information submitted by:
Activity Aerojet Engineering Corporation
Person M.B. Frankel and L.T. Carleton
Date 10 December, 1952

Preparation reaction(s):



1. Quantitative analysis: (% by weight)

	Carbon	Hydrogen	Oxygen	Nitrogen		
Calculated from formula	19.41	2.44	51.73	26.42		
By determination	20.18	2.53		26.44		

2. Burning properties: (compared to nitrocellulose, under nitrogen at atmospheric pressure:)

(faster? slower? residue? etc.)

3. Stability and Sensitivity: Plot any graphs on separate sheet

Discuss methods used when they vary from references. Give temperature used. (Use separate sheet if necessary.)

	Name of test	Recommended method	
a.	Impact Sensitivity	NOL	2.5-kg weight
b.	Thermal Stability	OSRD 3401 p.8	
c.	Vacuum Stability	NOL	48 hr (or less) at 100°C
d.	Temperature of Explosion	OSRD 3401 p.6	
e.	Temperature of Ignition	NOL	
f.			
g.	Impact Stability	Bureau of Mines Bull. No. 346, p. 72; 2-kg weight	
h.			

RESULTS OF ABOVE TESTS

	Reference compound (designation-TNT, Tetryl, N.C., etc.)	New Compound test results
a.	Tetryl, 32 cm/2.5 kg	19.6 cm/2.5 kg
b.		
c.		>30 cc/g in 2-1/2 hr
d.		
e.		173°C
f.		
g.	RDX, 30 cm/2 kg	25 cm/2 kg
h.		

4. Heat of formation: (ΔH) ± -39.8 (Indicate sign) Kg. calories at 25°C., 1 atm. pressure

Report No. 682
SPIA/M3

		By Experiment		By Calculation	Method (Description or reference. Separate sheet if necessary.)
5.	Energy of explosion (Q) (at 25°C, H_2O liquid)		cal/gm		
6.	Heat of combustion (H_c) (at 25°C, H_2O liquid)	2241	cal/gm	2232	Aerojet Report No. 417A
7.	Specific impulse (I_{sp}) calc:		lb-sec/lb		

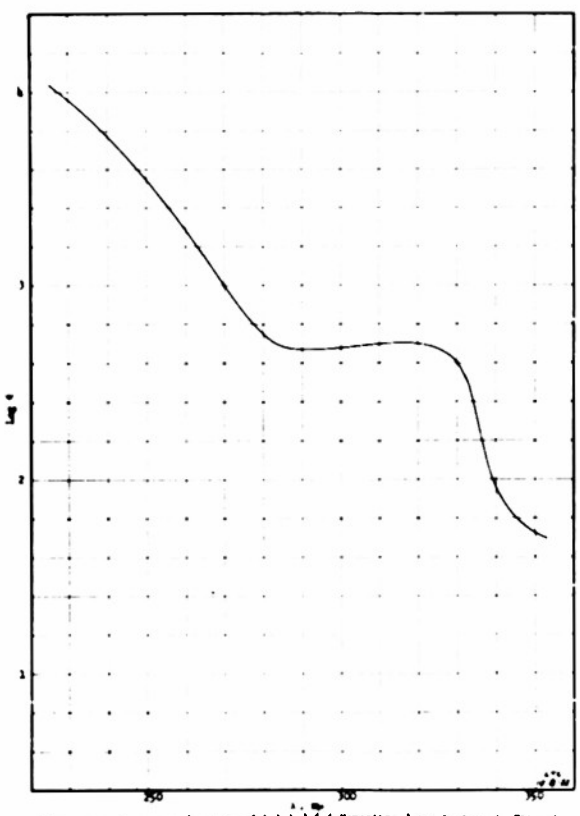
8. Physical form of compound (viscous liquid, crystalline type, etc.) Yellow, prismatic crystals

9. Simple microscope analysis data: (crystal studies)

10. Density (Macro method) 1.74 gm/cm³. (Micro or other method) ____ gm/cm³.
(Explain on separate sheet any unique methods you use.)

11. Index of refraction: ($n_D^{25°C}$) ____ 12. Color ____ 13. Odor none.

14. pH at 25°C. 3.7 (Method reference OSRD 3401 p.4, or OSRD 5968. Indicate method used, i.e. solvent and concentrations used. pH indicator paper or Beckman pH meter.) Beckman pH meter; 0.020M in acetone/water (5/1 volume ratio)



Ultraviolet Absorption Spectrum of 1,1,1,3,6,6-Hexanitro-3-aza-heptane in Ethanol

17. Boiling point, or decomposition temperature: decomposes on melting °C.
(underline which temperature is reported)

18. Heat of Vaporization: ____ g-cal./gm ____ BTU/lb.

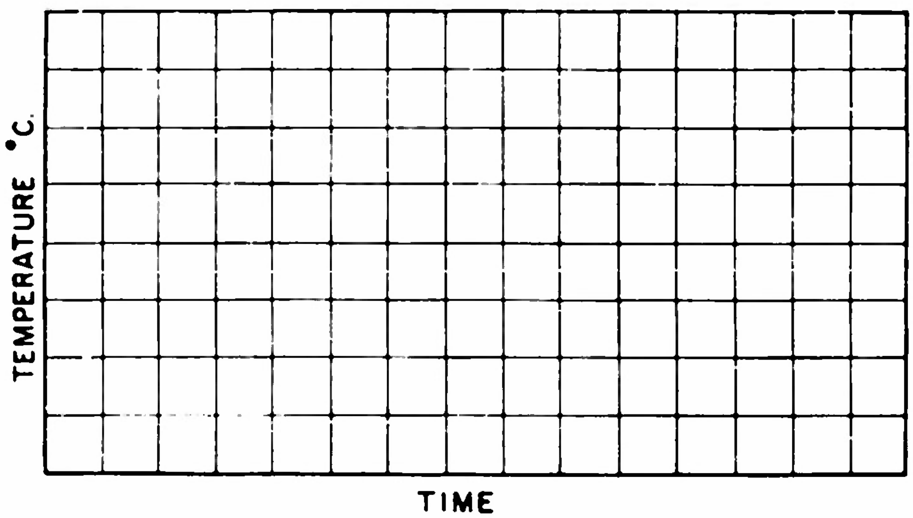
19. Heat of Fusion: ____ g-cal./gm ____ BTU/lb.

20. Melting point 105-107 °C.

Page 10
Appendix

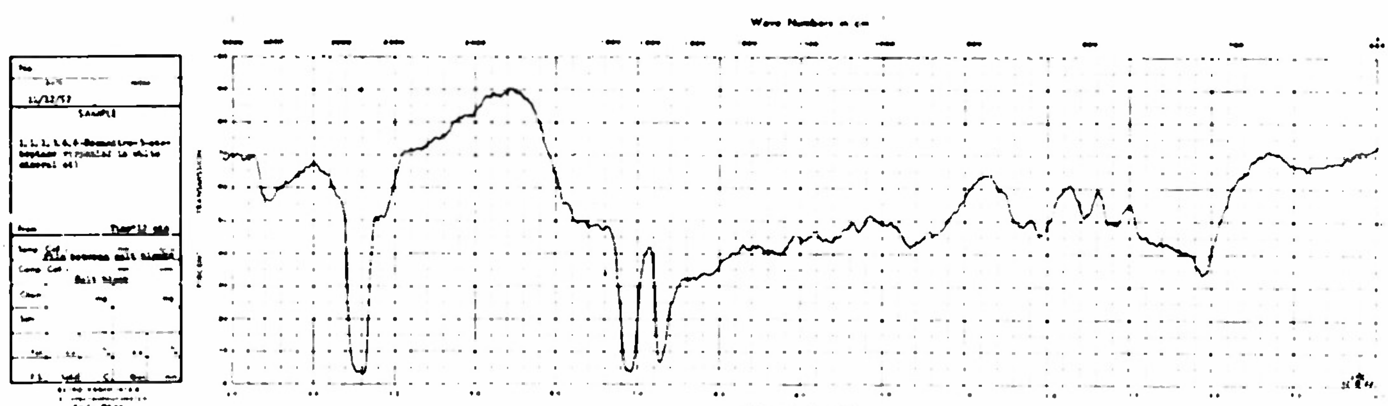
Report No. 682
SPIA/M3

21. Freezing point vs. Time Curve: (Please mark the coordinates with scale values that apply to the compound under study.)



22. Solubility of new compound:

<0.1	g/100 ml H_2O at 25°C.		g/100 ml H_2O at		°C.
120	g/100 ml	acetone. (name material used as solvent)	at	25	°C.
1.2	g/100 ml	toluene (name material used as solvent)	at	25	°C.



1,1,1,3,6,6-Hexanitro-3-aza-heptane Suspended in White Mineral Oil
Time=12 min

Under compatability we are considering the ability of two compounds to be in intimate contact (Note OSRD 5758 p. 21-22) over a long period of time without adverse effects on either the chemical or physical properties of either material. These tests will probably be of varied extent. They might be some of the following: (a) Standard stability tests. (b) Simple observations of exudation or separation at ambient or accelerated temperature. (c) Prepare thin sheets (.025" thick, 1" square) of the plasticized material. Separate the sheets with strips of cigarette paper or carbon paper and compress the stack in a "C" clamp. After several days note the oily collection on the paper. Please give reference to or describe procedure used. Item 28 is to indicate results when the compound is in contact with some material other than ethyl cellulose, nitrocellulose or rubber.

25. Compatability with ethyl cellulose: ____________________

26. Compatability with nitrocellulose: ____________________

27. Compatability with rubber: ____________________

28. Compatability with __________: ____________________

29. Polymerizing properties of the new compound:
   (a) By itself ____________________
   (b) In mixtures (with additives) ____________________
   (c) Inhibiting action on polymerization of:
      Thiokol ____________________
      Methacrylate ____________________
      Other compounds ____________________

30. Availability
   a. Amount now available? Research quantities
   b. When was available material first prepared? ____________________
   c. Amount prepared at that time? ____________________
   d. Is large production feasible? ____________________
   e. Plant capacity in existance, lbs/day? ____________________
   f. Outline steps for a quantity production method ____________________

31. Additional information: (toxicity, hazards, deterioration, oxygen balance, detonation rate, explosive power, ease of hydrolysis by water, etc. List references, reports, data books, etc. that refer to the compound.)

Calculated Lead Block Value = 162
Calculated Ballistic Mortar Value = 153 } Method of Aerojet Report No. 512, p. 8

Report No. 632
SPIA/M3

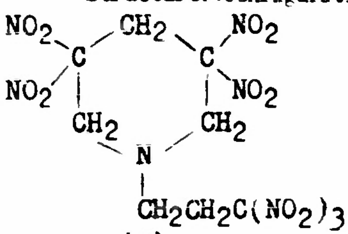
Data Questionnaire on

COMPOUNDS FOR USE AS INGREDIENTS OF PROPELLANTS AND OTHER EXPLOSIVES

Listed below are the characteristics which are believed to be of importance in the study of a compound which may possibly be used as a constituent of solid propellants or other explosives. It is not expected that all this information will be available for every compound. For example, with a compound showing poor stability, it is probably not advisable to investigate that compound farther. After placing such information as is available for a compound which you have prepared or tested on one of these forms, send it to SOLID PROPELLANT INFORMATION AGENCY, APL/JHU, 8621 Georgia Avenue, Silver Spring, Maryland. As additional information on the same or new compounds accrues, forward it on a similar form at a later date. The information submitted on these forms will be rewritten and published by SPIA in loose-leaf manual form. These forms may also be used as work or data sheets for your experimental studies. Extra copies are available upon request from SPIA. Suggestions for improvement of these forms are invited. If insufficient space has been provided for any item, attach separate sheets.

COMPOUND:
Name: N-3',3',3'-Trinitropropyl-3,3,5,5-tetranitropiperidine
Empirical formula: $C_8H_{10}N_8O_{14}$
Structure: (configuration)

Information submitted by:
Activity: Aerojet Engineering Corporation
Person: M.B. Frankel and L.T. Carleton
Date: 6 February, 1953



(I)

$$(NO_2)_3CCH_2CH_2NH_2\cdot HCl + \left[CH_2-\overset{NO_2}{\underset{NO_2}{C}}CH_2OH\right]_2 \xrightarrow{NaOH} I$$

1. Quantitative analysis: (% by weight)

	Carbon	Hydrogen	Oxygen	Nitrogen		
Calculated from formula	21.73	2.28	50.65	25.34		
By determination	21.33	2.29		24.52		

2. Burning properties: (compared to nitrocellulose, under nitrogen at atmospheric pressure:)

(faster? slower? residue? etc.)

3. Stability and Sensitivity: Plot any graphs on separate sheet. Discuss methods used when they vary from references. Give temperature used. (Use separate sheet if necessary.)

	Name of test	Recommended method	
a.	Impact Sensitivity	NOL ~~OSRD 3401~~	2.5-kg weight
b.	Thermal Stability	OSRD 3401 p.8	
c.	Vacuum Stability	NOL ~~OSRD 3401 p.13~~	48 hr at 100°C
d.	Temperature of Explosion	OSRD 3401 p.6	
e.	Temperature of Ignition	OSRD 3401 p.6	
f.	Impact Stability	Bureau of Mines Bull. No. 346, p. 72 (2-kg weight)	
g.			
h.			

RESULTS OF ABOVE TESTS

	Reference compound (designation-TNT, Tetryl, N.C., etc.)	New Compound test results
a.	Tetryl, 32 cm/2.5 kg	29 cm/2 kg
b.		
c.		>30 cc/g
d.		
e.		
f.	RDX, 30 cm/2 kg	25-35 cm/2 kg
g.		
h.		

4. Heat of formation: (ΔH) ± −60 (Indicate sign) Kg. calories at 25°C., 1 atm. pressure

Report No. 682
SPIA/M3

	By Experiment		By Calculation	Method (Description or reference. Separate sheet if necessary.)
5. Energy of explosion (Q) (at 25°C. H_2O liquid)		cal/gm		
6. Heat of combustion (H_c) (at 25°C. H_2O liquid)	2338	cal/gm	2392	Aerojet Report No. 417A
7. Specific impulse (I_{sp}) calc:		lb-sec/lb		

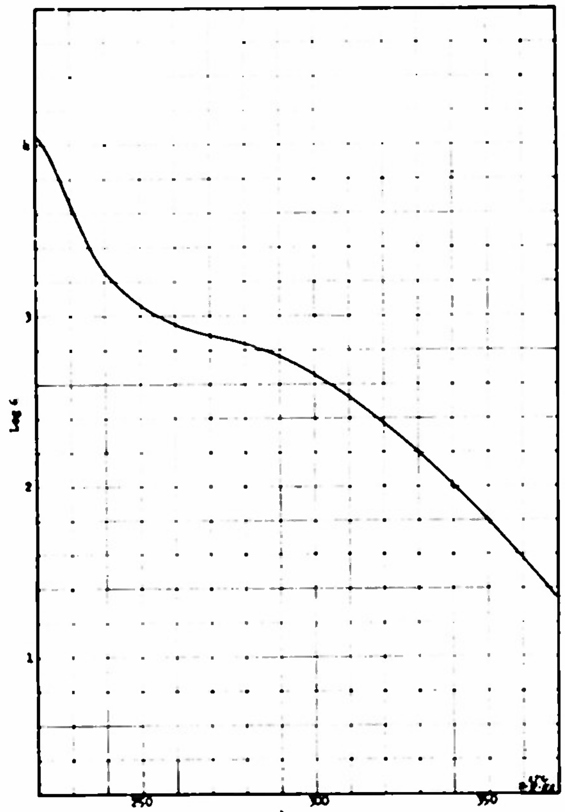
8. Physical form of compound (viscous liquid, crystalline type, etc.) Prismatic white crystals

9. Simple microscope analysis data: (crystal studies)

10. Density (Micro method) 1.703 gm/cm³. (Micro or other method) ________ gm/cm³. (Explain on separate sheet any unique methods you use.)

11. Index of refraction: ($n_D^{25°C.}$) ________ 12. Color white 13. Odor acid.

14. pH at 25°C. 2.4 (Method reference OSRD 3401 p.4, or OSRD 5968. Indicate method used, i.e. solvent and concentrations used, pH indicator paper or Beckman pH meter.) with Beckman pH meter in 0.034M acetone/water (5/1 volume ratio)



N-3',3',3'-Trinitropropyl-3,3,5,5-tetranitropiperidine in Methanol

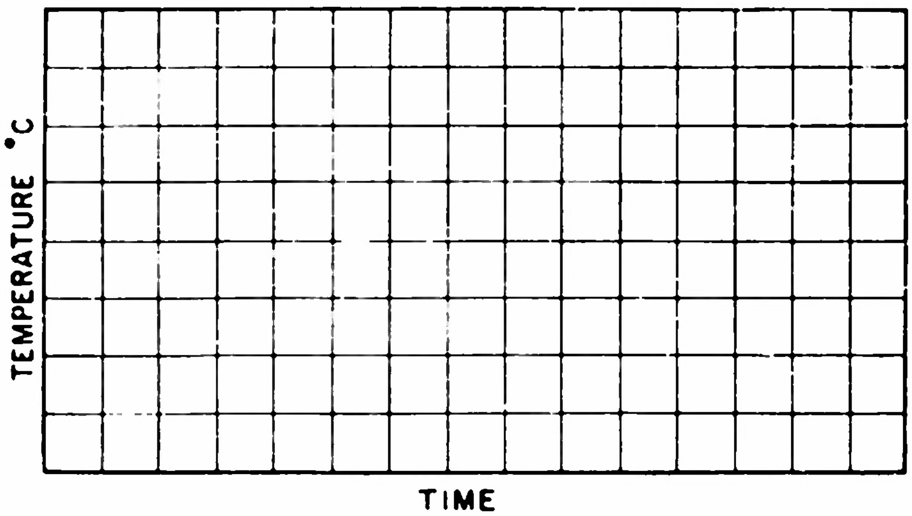
17. Boiling point, or decomposition temperature: decomposes on melting °C. (underline which temperature is reported)

18. Heat of Vaporization: ________ g-cal./gm ________ BTU/lb.

19. Heat of Fusion: ________ g-cal./gm ________ BTU/lb.

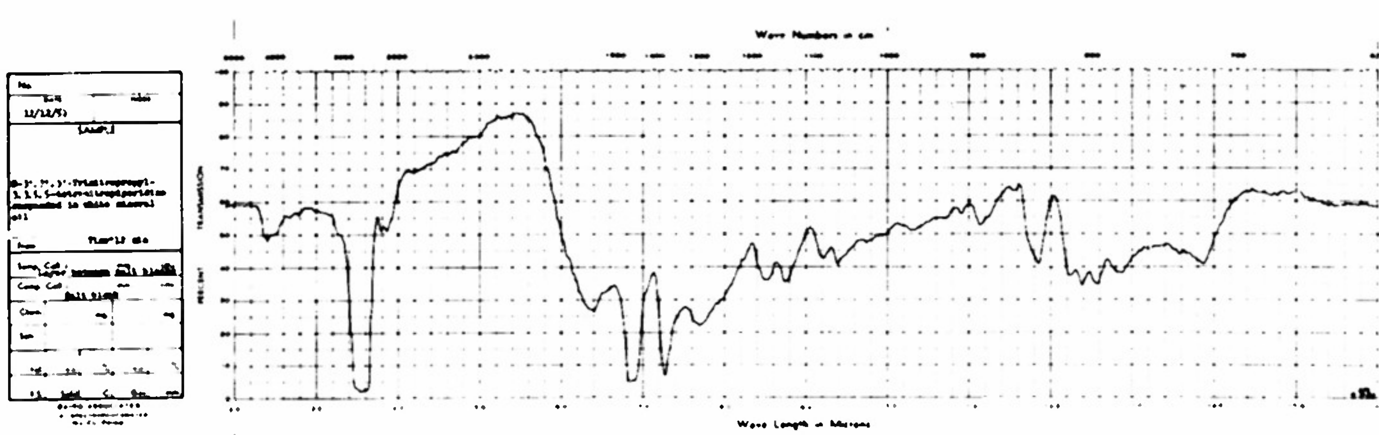
20. Melting point 144-146 °C. (d)

21. Freezing point vs. Time Curve: (Please mark the coordinates with scale values that apply to the compound under study.)



22. Solubility of new compound:

<0.1	g/100 ml H_2O at 25°C.		g/100 ml H_2O at		°C.
3.5	g/100 ml	toluene (name material used as solvent)	at	25	°C.
220	g/100 ml	acetone (name material used as solvent)	at	25	°C.



N-3',3',3'-Trinitropropyl-3,3,5,5-tetranitropiperidine
Suspended in White Mineral Oil
Time=12 min

Under compatability we are considering the ability of two compounds to be in intimate contact (Note OSRD 5758 p. 21-22) over a long period of time without adverse effects on either the chemical or physical properties of either material. These tests will probably be of varied extent. They might be some of the following: (a) Standard stability tests. (b) Simple observations of exudation or separation at ambient or accelerated temperature. (c) Prepare thin sheets (.025" thick, 1" square) of the plasticized material. Separate the sheets with strips of cigarette paper or carbon paper and compress the stack in a "C" clamp. After several days note the oily collection on the paper. Please give reference to or describe procedure used. Item 28 is to indicate results when the compound is in contact with some material other than ethyl cellulose, nitrocellulose or rubber.

25. Compatability with ethyl cellulose: __________

26. Compatability with nitrocellulose: __________

27. Compatability with rubber: __________

28. Compatability with __________: __________

29. Polymerizing properties of the new compound:
   (a) By itself __________
   (b) In mixtures (with additives) __________
   (c) Inhibiting action on polymerization of:
      Thiokol __________
      Methacrylate __________
      Other compounds __________

30. Availability
   a. Amount now available? Research quantities
   b. When was available material first prepared? __________
   c. Amount prepared at that time? __________
   d. Is large production feasible? __________
   e. Plant capacity in existance, lbs/day? __________
   f. Outline steps for a quantity production method __________

31. Additional information: (toxicity, hazards, deterioration, oxygen balance, detonation rate, explosive power, ease of hydrolysis by water, etc. List references, reports, data books, etc. that refer to the compound.)

Calculated Lead Block Value = 155
Calculated Ballistic Mortar Value = 152

$\Big\}$ Method of Aerojet Report No. 512, p. 8

Report No. 682
SPIA/M3

Data Questionnaire on

COMPOUNDS FOR USE AS INGREDIENTS OF PROPELLANTS AND OTHER EXPLOSIVES

Listed below are the characteristics which are believed to be of importance in the study of a compound which may possibly be used as a constituent of solid propellants or other explosives. It is not expected that all this information will be available for every compound. For example, with a compound showing poor stability, it is probably not advisable to investigate that compound farther. After placing such information as is available for a compound which you have prepared or tested on one of these forms, send it to SOLID PROPELLANT INFORMATION AGENCY, APL/JHU, 8621 Georgia Avenue, Silver Spring, Maryland. As additional information on the same or new compounds accrues, forward it on a similar form at a later date. The information submitted on these forms will be rewritten and published by SPIA in loose-leaf manual form. These forms may also be used as work or data sheets for your experimental studies. Extra copies are available upon request from SPIA. Suggestions for improvement of these forms are invited. If insufficient space has been provided for any item, attach separate sheets.

COMPOUND:
Name 3,3,3-Trinitropropyl Amine Nitrate
Empirical formula $C_3H_7N_5O_9$
Structure: (configuration)

$(NO_2)_3CCH_2CH_2NH_2 \cdot HNO_3$

Information submitted by:
Activity Aerojet Engineering Corporation
Person M.B. Frankel and L.T. Carleton
Date 6 February, 1953

$$(NO_2)_3CCH_2CH_2NCO + HNO_3 + H_2O \longrightarrow (NO_2)_3CCH_2CH_2NH_2 \cdot HNO_3 + CO_2$$

Preparation reaction(s):

1. Quantitative analysis: (% by weight)

	Carbon	Hydrogen	Oxygen	Nitrogen		
Calculated from formula	14.01	2.74	56.01	27.24		
By determination	14.09	2.80		26.58		

2. Burning properties: (compared to nitrocellulose, under nitrogen at atmospheric pressure:)

(faster? slower? residue? etc.)

3. Stability and Sensitivity: Plot any graphs on separate sheet

Discuss methods used when they vary from references. Give temperature used. (Use separate sheet if necessary.)

	Name of test	Recommended method	
a.	Impact Sensitivity	NOL ~~[illegible]~~	2.5-kg weight
b.	Thermal Stability	OSRD 3401 p.8	
c.	Vacuum Stability	NOL ~~[illegible]~~	48 hr at 100°C
d.	Temperature of Explosion	OSRD 3401 p.6	
e.	Temperature of Ignition	OSRD 3401 p.6	
f.	Impact Stability	Bureau of Mines Bull.	No. 346, p. 72 (2-kg weight)
g.			
h.			

RESULTS OF ABOVE TESTS

	Reference compound (designation-TNT, Tetryl, N.C., etc.)	New Compound test results
a.	Tetryl, 32 cm/2.5 kg	10 cm/2.5 kg
b.		
c.		>30 cc/g
d.		
e.		
f.	RDX, 30 cm/2 kg	30-40 cm/2 kg
g.		
h.		

4. Heat of formation: (ΔH) ± -95 (Indicate sign) Kg. calories at 25°C., 1 atm. pressure

Report No. 682
SPIA/M3

	By Experiment		By Calculation	Method (Description or reference. Separate sheet if necessary.)
5. Energy of explosion (Q) (at 25°C, H_2O liquid)		cal/gm		
6. Heat of combustion (H_c) (at 25°C, H_2O liquid)	1657	cal/gm	1706	Aerojet Report No. 417A
7. Specific impulse (I_{sp}) calc:		lb-sec/lb		

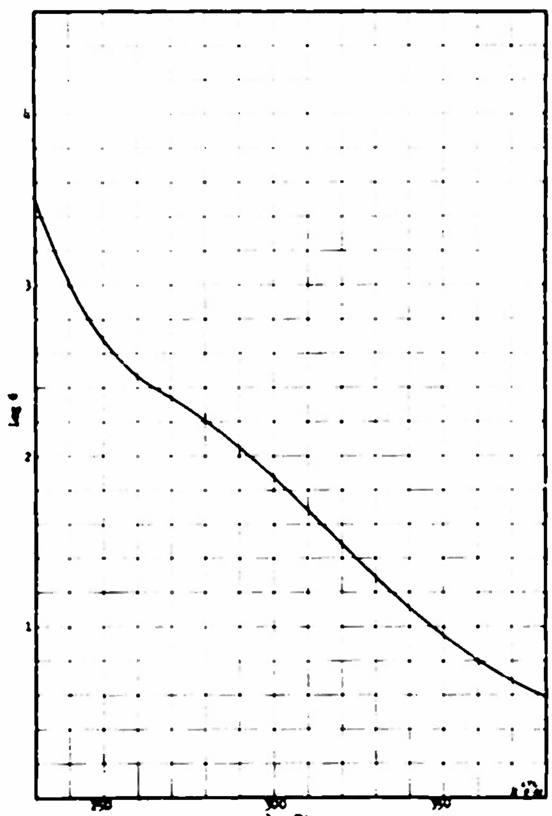
8. Physical form of compound (viscous liquid, crystalline type, etc.) White crystals

9. Simple microscope analysis data: (crystal studies)

10. Density (Macro method) 1.774 gm/cm³. (Micro or other method) ______ gm/cm³. (Explain on separate sheet any unique methods you use.)

11. Index of refraction: ($n_D^{25°C.}$) ______ 12. Color white 13. Odor none.

14. pH at 25°C. 3.3 (Method reference OSRD 3401 p.4, or OSRD 5968. Indicate method used, i.e. solvent and concentrations used. pH indicator paper or Beckman pH meter.) with Beckman pH meter in 0.031M acetone/water (5/1 volume ratio)



1,1,3-Trinitropropyl Amine Nitrate in Ethanol

17. Boiling point, or decomposition temperature: (underline which temperature is reported) decomposes on melting °C.

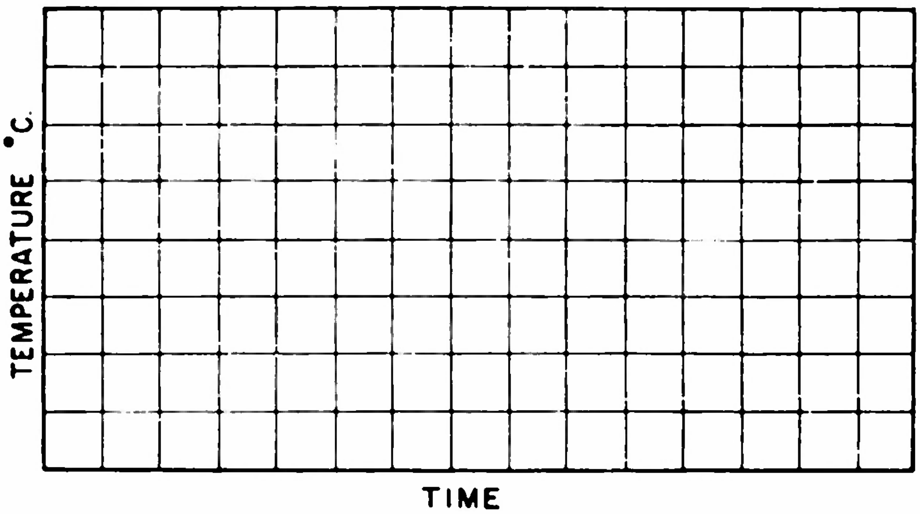
18. Heat of Vaporization: ______ g-cal./gm ______ BTU/lb.

19. Heat of Fusion: ______ g-cal./gm ______ BTU/lb.

20. Melting point 132-134 °C. (d)

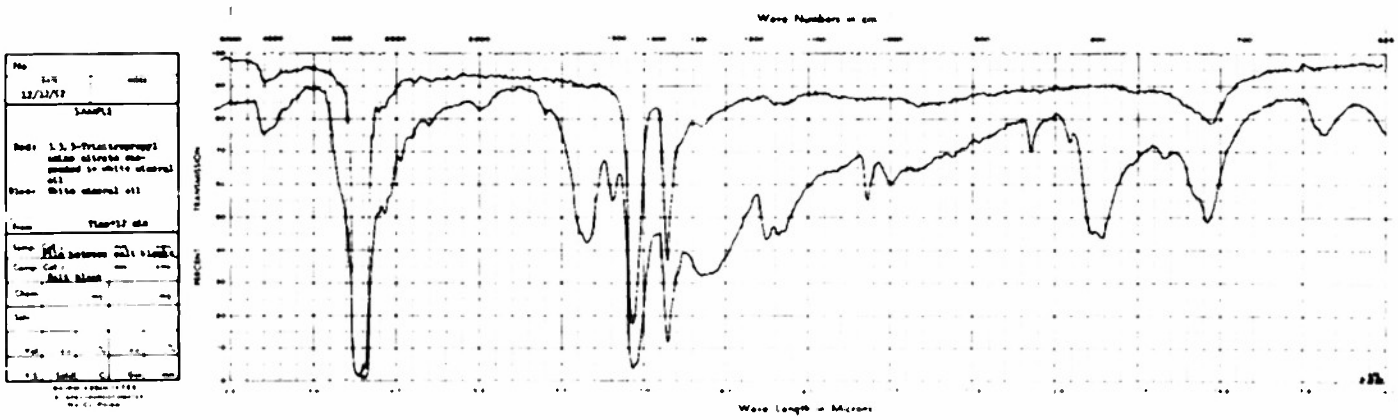
Report No. 682
SPIA/M3

21. Freezing point vs. Time Curve: (Please mark the coordinates with scale values that apply to the compound under study.)



22. Solubility of new compound:

32	g/100 ml H_2O at 25°C.	______ g/100 ml H_2O at	______	°C.
<0.1	g/100 ml	toluene (name material used as solvent)	at 25	°C.
170	g/100 ml	acetone (name material used as solvent)	at 25	°C.



Red: 3,3,3-Trinitropropyl Amine Nitrate Suspended in White Mineral Oil
Blue: White Mineral Oil
Time=12 min

Under compatability we are considering the ability of two compounds to be in intimate contact (Note OSRD 5758 p. 21-22) over a long period of time without adverse effects on either the chemical or physical properties of either material. These tests will probably be of varied extent. They might be some of the following: (a) Standard stability tests. (b) Simple observations of exudation or separation at ambient or accelerated temperature. (c) Prepare thin sheets (.025" thick, 1" square) of the plasticized material. Separate the sheets with strips of cigarette paper or carbon paper and compress the stack in a "C" clamp. After several days note the oily collection on the paper. Please give reference to or describe procedure used. Item 28 is to indicate results when the compound is in contact with some material other than ethyl cellulose, nitrocellulose or rubber.

25. **Compatability with ethyl cellulose:** ______

26. **Compatability with nitrocellulose:** ______

27. **Compatability with rubber:** ______

28. **Compatability with** ______ **:** ______

29. **Polymerizing properties of the new compound:**
   (a) **By itself** ______
   (b) **In mixtures** (with additives) ______
   (c) **Inhibiting action on polymerization of:**
      Thiokol ______
      Methacrylate ______
      Other compounds ______

30. **Availability**
   a. **Amount now available?** Research quantities
   b. **When was available material first prepared?** ______
   c. **Amount prepared at that time?** ______
   d. **Is large production feasible?** ______
   e. **Plant capacity in existance, lbs/day?** ______
   f. **Outline steps for a quantity production method** ______

31. **Additional information:** (toxicity, hazards, deterioration, oxygen balance, detonation rate, explosive power, ease of hydrolysis by water, etc. List references, reports, data books, etc. that refer to the compound.)

Calculated Lead Block Value = 171
Calculated Ballistic Mortar Value = 144
Method of Aerojet Report No. 517, p. 8

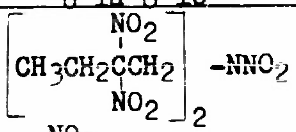
Report No. 682
SPIA/M3

Date Questionnaire on

COMPOUNDS FOR USE AS INGREDIENTS OF PROPELLANTS AND OTHER EXPLOSIVES

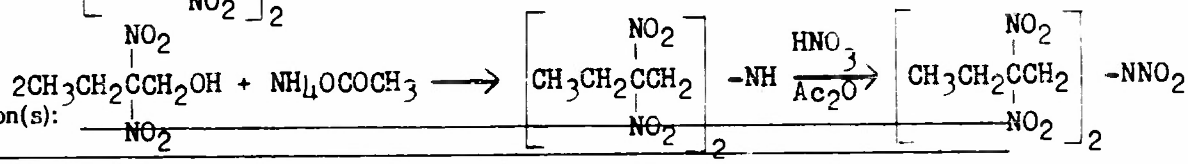
Listed below are the characteristics which are believed to be of importance in the study of a compound which may possibly be used as a constituent of solid propellants or other explosives. It is not expected that all this information will be available for every compound. For example, with a compound showing poor stability, it is probably not advisable to investigate that compound farther. After placing such information as is available for a compound which you have prepared or tested on one of these forms, send it to SOLID PROPELLANT INFORMATION AGENCY, APL/JHU, 8621 Georgia Avenue, Silver Spring, Maryland. As additional information on the same or new compounds accrues, forward it on a similar form at a later date. The information submitted on these forms will be rewritten and published by SPIA in loose-leaf manual form. These forms may also be used as work or data sheets for your experimental studies. Extra copies are available upon request from SPIA. Suggestions for improvement of these forms are invited. If insufficient space has been provided for any item, attach separate sheets.

COMPOUND:
Name 3,3,5,7,7-Pentanitro-5-aza-nonane
Empirical formula $C_8H_{14}N_6O_{10}$
Structure: (configuration)



Information submitted by:
Activity Aerojet Engineering Corporation
Person M.B. Frankel and L.T. Carleton
Date 6 February, 1953

Preparation reaction(s):



1. Quantitative analysis: (% by weight)

	Carbon	Hydrogen	Oxygen	Nitrogen		
Calculated from formula	27.12	3.98	45.17	23.73		
By determination	27.17	3.72		22.74		

2. Burning properties: (compared to nitrocellulose, under nitrogen at atmospheric pressure:)

(faster? slower? residue? etc.)

3. Stability and Sensitivity: Plot any graphs on separate sheet. Discuss methods used when they vary from references. Give temperature used. (Use separate sheet if necessary.)

	Name of test	Recommended method	
a.	Impact Sensitivity	NOL ~~[illegible]~~	2.5-kg weight
b.	Thermal Stability	OSRD 3401 p.8	
c.	Vacuum Stability	NOL ~~OSRD 3401 p.10~~	48 hr at 100°C
d.	Temperature of Explosion	OSRD 3401 p.6	
e.	Temperature of Ignition	OSRD 3401 p.6	
f.	Impact Stability	Bureau of Mines Bull.	No. 346, p. 72 (2-kg weight)
g.			
h.			

RESULTS OF ABOVE TESTS

	Reference compound (designation-TNT, Tetryl, N.C., etc.)	New Compound test results
a.	Tetryl, 32 cm/2 kg	80 cm/2.5 kg
b.		
c.		1.64 cc/g
d.		
e.		
f.	RDX, 30 cm/2 kg	25-30 cm/2 kg
g.		
h.		

4. Heat of formation: (ΔH) ± -85 (Indicate sign) Kg. calories at 25°C., 1 atm. pressure

	By Experiment		By Calculation	Method Description or reference. Separate sheet if necessary.
5. Energy of explosion (Q) (at 25°C. H_2O liquid)		cal/gm		
6. Heat of combustion (H_c) (at 25°C. H_2O liquid)	3234	cal/gm	3290	Aerojet Report No. 417A
7. Specific impulse (I_{sp}) calc:		lb-sec/lb		

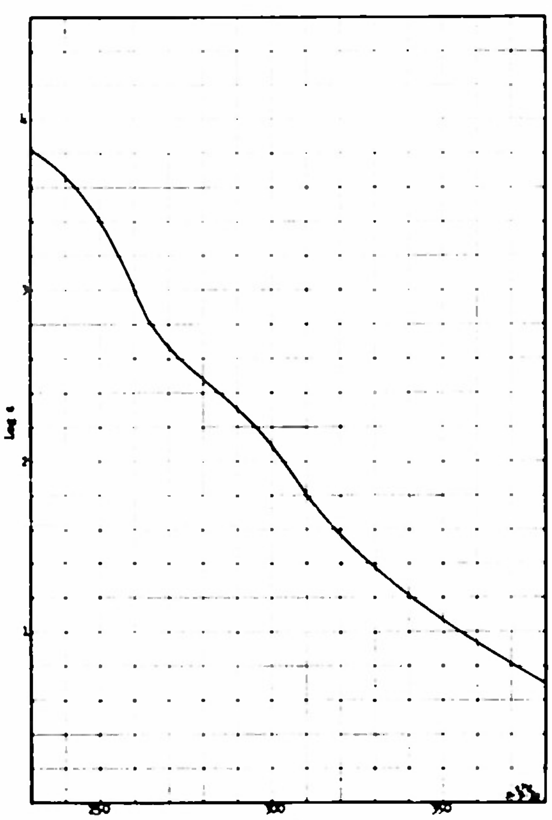
8. Physical form of compound (viscous liquid, crystalline type, etc.) Flaky white crystals

9. Simple microscope analysis data: (crystal studies)

10. Density (Macro method) 1.707 gm/cm³. (Micro or other method) ____ gm/cm³. (Explain on separate sheet any unique methods you use.)

11. Index of refraction: ($n_D^{25°C.}$) ____ 12. Color white 13. Odor none.

14. pH at 25°C. 5.8 (Method reference OSRD 3401 p.4, or OSRD 5968. Indicate method used, i.e. solvent and concentrations used. pH indicator paper or Beckman pH meter.) with Beckman pH meter in 0.041M acetone/water (5/1 volume ratio)



3,3,5,7,7-Pentanitro-5-aza-nonane in Methanol

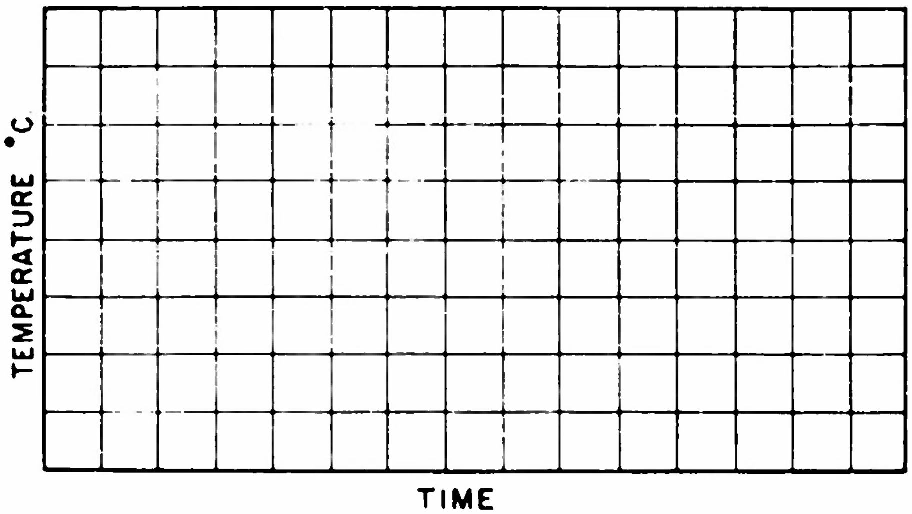
17. Boiling point, or decomposition temperature: ____ ____ °C. (underline which temperature is reported)

18. Heat of Vaporization: ____ g-cal./gm ____ BTU/lb.

19. Heat of Fusion: ____ g-cal./gm ____ BTU/lb.

20. Melting point 107-108 °C.

21. Freezing point vs. Time Curve: (Please mark the coordinates with scale values that apply to the compound under study.)

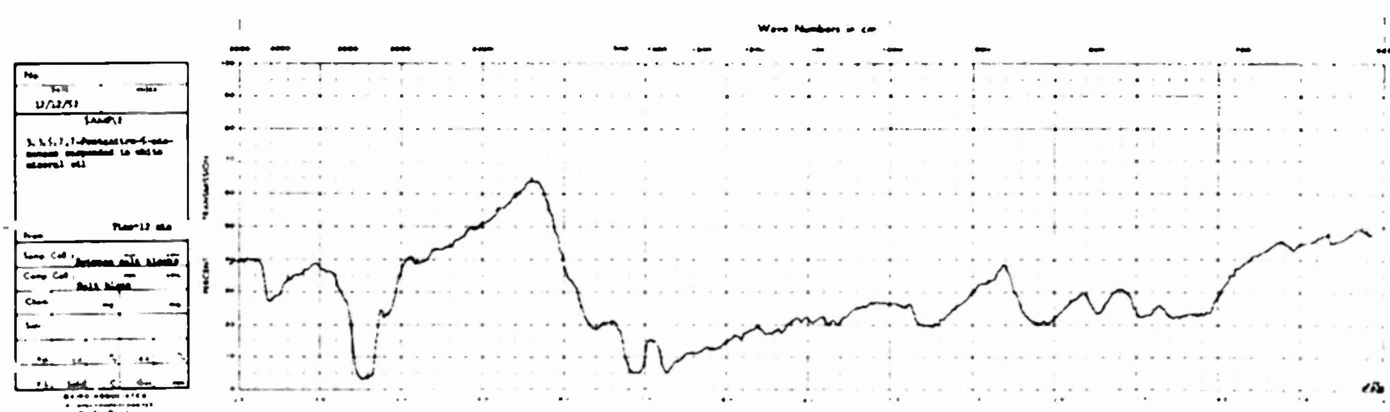


22. Solubility of new compound:

<0.1 g/100 ml H_2O at 25°C. ______ g/100 ml H_2O at ______ °C.

2.3 g/100 ml toluene at 25 °C.
(name material used as solvent)

67 g/100 ml acetone at 25 °C.
(name material used as solvent)



3,3,5,7,7-Pentanitro-5-aza-nonane Suspended in White Mineral Oil
Time=12 min

Report No. 682
SPIA/M3

Under compatability we are considering the ability of two compounds to be in intimate contact (Note OSRD 5758 p. 21-22) over a long period of time without adverse effects on either the chemical or physical properties of either material. These tests will probably be of varied extent. They might be some of the following: (a) Standard stability tests. (b) Simple observations of exudation or separation at ambient or accelerated temperature. (c) Prepare thin sheets (.025" thick, 1" square) of the plasticized material. Separate the sheets with strips of cigarette paper or carbon paper and compress the stack in a "C" clamp. After several days note the oily collection on the paper. Please give reference to or describe procedure used. Item 28 is to indicate results when the compound is in contact with some material other than ethyl cellulose, nitrocellulose or rubber.

25. Compatability with ethyl cellulose: __________

26. Compatability with nitrocellulose: __________

27. Compatability with rubber: __________

28. Compatability with __________: __________

29. Polymerizing properties of the new compound:
   (a) By itself
   (b) In mixtures (with additives)
   (c) Inhibiting action on polymerization of:
      Thiokol
      Methacrylate
      Other compounds

30. Availability
   a. Amount now available? Research quantities
   b. When was available material first prepared?
   c. Amount prepared at that time?
   d. Is large production feasible?
   e. Plant capacity in existance, lbs/day?
   f. Outline steps for a quantity production method

31. Additional information: (toxicity, hazards, deterioration, oxygen balance, detonation rate, explosive power, ease of hydrolysis by water, etc. List references, reports, data books, etc. that refer to the compound.)

$\left.\begin{array}{l}\text{Calculated Lead Block Value = 112}\\ \text{Calculated Ballistic Mortar Value = 129}\end{array}\right\}$ Method of Aerojet Report No. 512, p. 8

Report No. 682
SPIA/M3

Data Questionnaire on

COMPOUNDS FOR USE AS INGREDIENTS OF PROPELLANTS AND OTHER EXPLOSIVES

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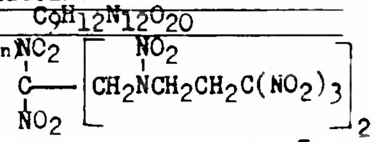
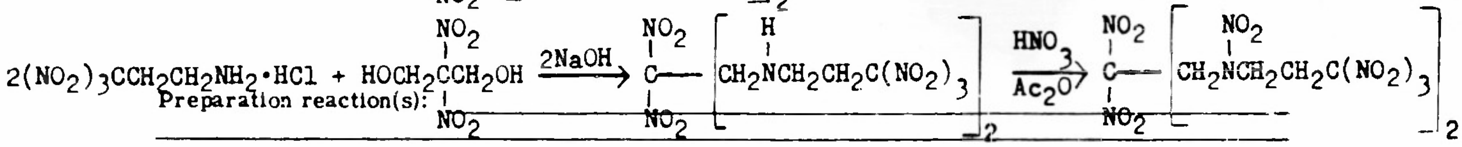
COMPOUND:
Name 1,1,1,4,6,6,8,11,11,11-Decanitro-4,8-diaza-undecane
Empirical formula $C_9H_{12}N_{12}O_{20}$
Structure: (configuration)



Information submitted by:
Activity Aerojet Engineering Corporation
Person M.B. Frankel and L.T. Carleton
Date 6 February 1953

Preparation reaction(s):



1. Quantitative analysis: (% by weight)

	Carbon	Hydrogen	Oxygen	Nitrogen		
Calculated from formula	17.77	1.99	52.60	27.64		
By determination	18.29	2.02		27.40		

2. Burning properties: (compared to nitrocellulose, under nitrogen at atmospheric pressure:)

(faster? slower? residue? etc.)

3. Stability and Sensitivity: Plot any graphs on separate sheet

Discuss methods used when they vary from references. Give temperature used. (Use separate sheet if necessary.)

	Name of test	Recommended method	
a.	Impact Sensitivity	NOL ~~[illegible]~~	2.5-kg weight
b.	Thermal Stability	OSRD 3401 p.8	
c.	Vacuum Stability	NOL ~~[illegible]~~	48 hr at 100°C
d.	Temperature of Explosion	OSRD 3401 p.6	
e.	Temperature of Ignition	OSRD 3401 p.6	
f.	Impact Stability	Bureau of Mines Bull.	No. 346, p. 72 (2-kg weight)
g.			
h.			

RESULTS OF ABOVE TESTS

	Reference compound (designation-TNT, Tetryl, N.C., etc.)	New Compound test results
a.	Tetryl, 32 cm/2.5 kg	10 cm/2.5 kg
b.		
c.		1.87 cc/g
d.		
e.		
f.	RDX, 30 cm/2 kg	5-10 cm/2 kg
g.		
h.		

4. Heat of formation: (ΔH) ± -50 (Indicate sign) Kg. calories at 25°C., 1 atm. pressure

Report No. 682
SPIA/M3

		By Experiment		By Calculation	Method (Description or reference. Separate sheet if necessary.)
5.	Energy of explosion (Q) (at 25°C. H_2O liquid)		cal/gm		
6.	Heat of combustion (H_c) (at 25°C. H_2O liquid)	1983	cal/gm	2044	Aerojet Report No. 417A
7.	Specific impulse (I_{sp}) calc:		lb-sec/lb		

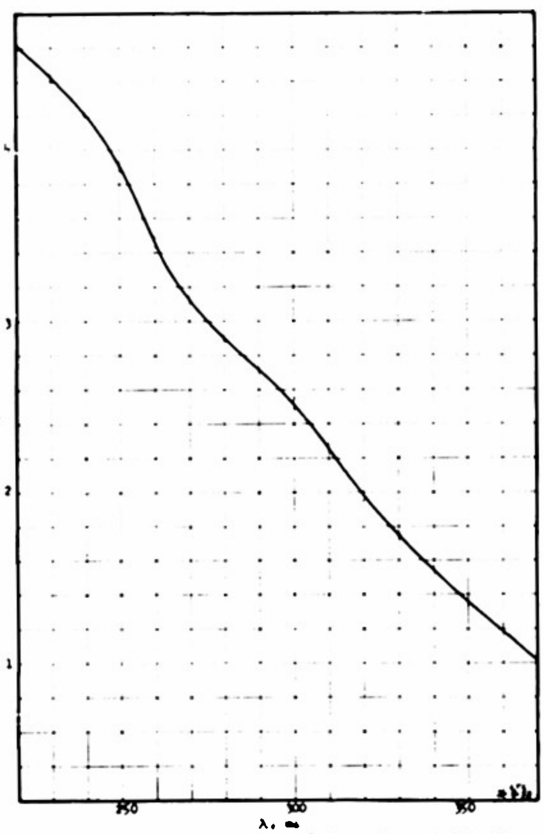
8. Physical form of compound (viscous liquid, crystalline type, etc.) White crystals

9. Simple microscope analysis data: (crystal studies)

10. Density (Macro method) 1.81 gm/cm³. (Micro or other method) ______ gm/cm³. (Explain on separate sheet any unique methods you use.)

11. Index of refraction: ($n_D^{25°C.}$) ______ 12. Color white 13. Odor none.

14. pH at 25°C. 6.5 (Method reference OSRD 3401 p.4, or OSRD 5968. Indicate method used, i.e. solvent and concentrations used, pH indicator paper or Beckman pH meter.) with Beckman pH meter, 0.0071M in acetone/water (5/1 volume ratio)



1,1,1,4,6,6,8,11,11,11-Decanitro-4,8-diaza-undecane in Ethanol

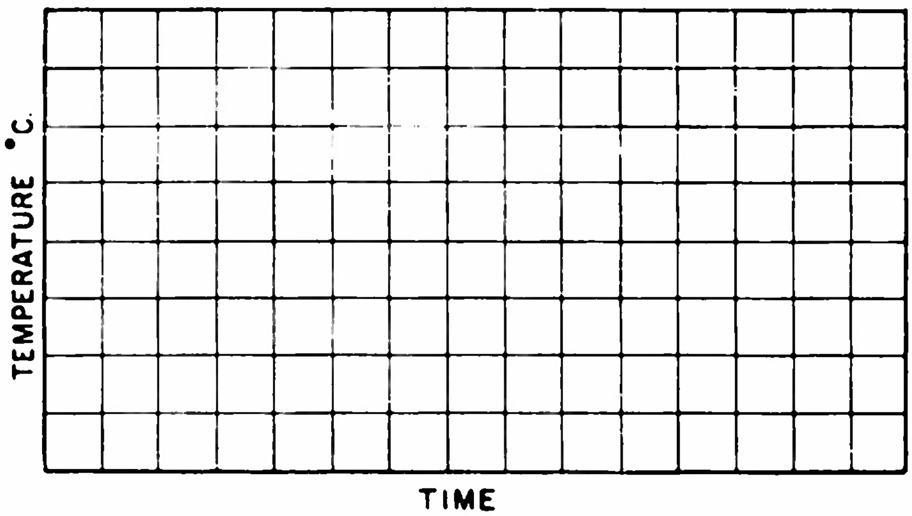
17. Boiling point, or decomposition temperature: decomposes on melting °C. (underline which temperature is reported)

18. Heat of Vaporization: ______ g-cal./gm ______ BTU/lb.

19. Heat of Fusion: ______ g-cal./gm ______ BTU/lb.

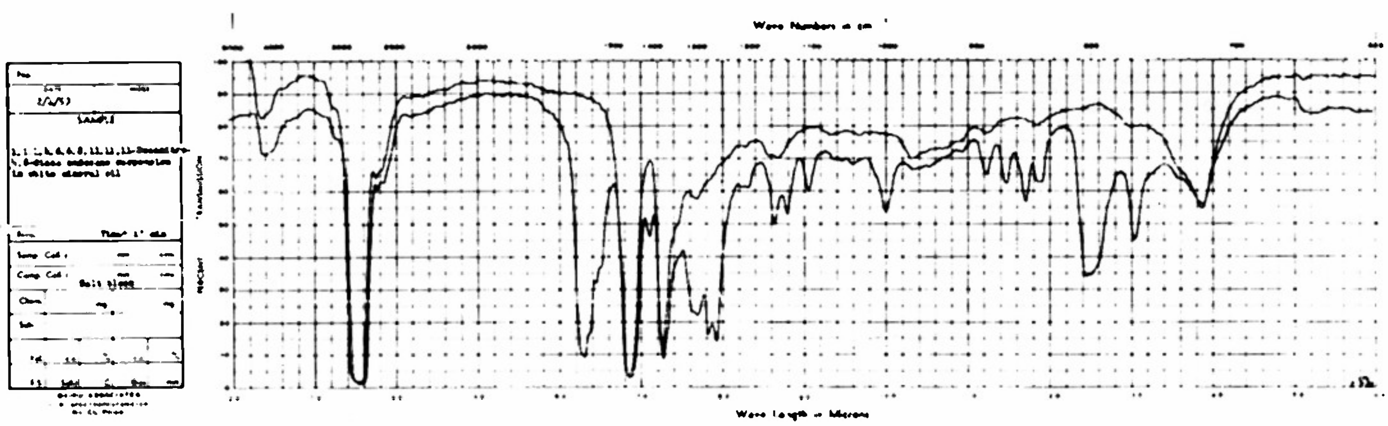
20. Melting point 157-159 °C.(d)

21. Freezing point vs. Time Curve: (Please mark the coordinates with scale values that apply to the compound under study.)



22. Solubility of new compound:

<0.1	g/100 ml H_2O at 25°C.		g/100 ml H_2O at		°C.
<0.1	g/100 ml	toluene (name material used as solvent)	at	25	°C.
110	g/100 ml	acetone (name material used as solvent)	at	25	°C.



1,1,1,4,6,6,8,11,11,11-Decanitro-4,8-diaza Undecane Suspension in White Mineral Oil
Time=12 min

Report No. 682
SPIA/M3

Under compatability we are considering the ability of two compounds to be in intimate contact (Note OSRD 5758 p. 21-22) over a long period of time without adverse effects on either the chemical or physical properties of either material. These tests will probably be of varied extent. They might be some of the following: (a) Standard stability tests. (b) Simple observations of exudation or separation at ambient or accelerated temperature. (c) Prepare thin sheets (.025" thick, 1" square) of the plasticized material. Separate the sheets with strips of cigarette paper or carbon paper and compress the stack in a "C" clamp. After several days note the oily collection on the paper. Please give reference to or describe procedure used. Item 28 is to indicate results when the compound is in contact with some material other than ethyl cellulose, nitrocellulose or rubber.

25. Compatability with ethyl cellulose:

26. Compatability with nitrocellulose:

27. Compatability with rubber:

28. Compatability with ____________:

29. Polymerizing properties of the new compound:
- (a) By itself
- (b) In mixtures (with additives)
- (c) Inhibiting action on polymerization of:
  - Thiokol
  - Methacrylate
  - Other compounds

30. Availability
- a. Amount now available? Research quantities
- b. When was available material first prepared?
- c. Amount prepared at that time?
- d. Is large production feasible?
- e. Plant capacity in existance, lbs/day?
- f. Outline steps for a quantity production method

31. Additional information: (toxicity, hazards, deterioration, oxygen balance, detonation rate, explosive power, ease of hydrolysis by water, etc. List references, reports, data books, etc. that refer to the compound.)

Calculated Lead Block Value = 177
Calculated Ballistic Mortar Value = 155

Method of Aerojet Report No. 512, p. 8

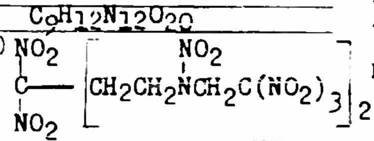
Report No. 682
SPIA/M3

Data Questionnaire on

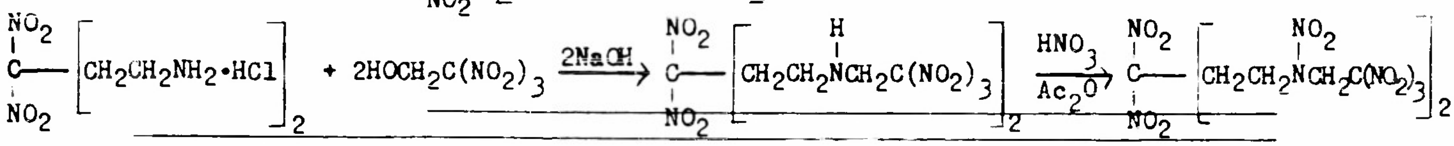
COMPOUNDS FOR USE AS INGREDIENTS OF PROPELLANTS AND OTHER EXPLOSIVES

Listed below are the characteristics which are believed to be of importance in the study of a compound which may possibly be used as a constituent of solid propellants or other explosives. It is not expected that all this information will be available for every compound. For example, with a compound showing poor stability, it is probably not advisable to investigate that compound farther. After placing such information as is available for a compound which you have prepared or tested on one of these forms, send it to SOLID PROPELLANT INFORMATION AGENCY, APL/JHU, 8621 Georgia Avenue, Silver Spring, Maryland. As additional information on the same or new compounds accrues, forward it on a similar form at a later date. The information submitted on these forms will be rewritten and published by SPIA in loose-leaf manual form. These forms may also be used as work or data sheets for your experimental studies. Extra copies are available upon request from SPIA. Suggestions for improvement of these forms are invited. If insufficient space has been provided for any item, attach separate sheets.

COMPOUND: Name 1,1,1,3,6,6,9,11,11,11-Decanitro-3,9-diaza-undecane
Empirical formula $C_9H_{12}N_{12}O_{20}$
Structure: (configuration)



Information submitted by:
Activity Aerojet Engineering Corporation
Person M.B. Frankel and L.T. Carleton
Date 6 February 1953



1. Quantitative analysis: (% by weight)

	Carbon	Hydrogen	Oxygen	Nitrogen		
Calculated from formula	17.77	1.99	52.60	27.64		
By determination	18.11	2.00		27.34		

2. Burning properties: (compared to nitrocellulose, under nitrogen at atmospheric pressure:)

(faster? slower? residue? etc.)

3. Stability and Sensitivity: Plot any graphs on separate sheet. Discuss methods used when they vary from references. Give temperature used. (Use separate sheet if necessary.)

	Name of test	Recommended method	
a.	Impact Sensitivity	NOL	2.5-kg weight
b.	Thermal Stability	OSRD 3401 p.8	
c.	Vacuum Stability	NOL	48 hr at 100°C
d.	Temperature of Explosion	OSRD 3401 p.6	
e.	Temperature of Ignition	OSRD 3401 p.6	
f.	Impact Stability	Bureau of Mines Bull. No. 346, p. 72 (2-kg weight)	
g.			
h.			

RESULTS OF ABOVE TESTS

	Reference compound (designation-TNT, Tetryl, N.C., etc.)	New Compound test results
a.	Tetryl, 32 cm/2.5 kg	11 cm/2.5 kg
b.		
c.		20.6 cc/g
d.		
e.		
f.	RDX, 30 cm/2 kg	10-15 cm/2 kg
g.		
h.		

4. Heat of formation: (ΔH) ± -60 (indicate sign) Kg. calories at 25°C., 1 atm. pressure

Report No. 682
SPIA/M3

		By Experiment		By Calculation	Method (Description or reference. Separate sheet if necessary.)
5.	Energy of explosion (Q) (at 25°C, H_2O liquid)		cal/gm		
6.	Heat of combustion (H_c) (at 25°C, H_2O liquid)	1967	cal/gm	2044	Aerojet Report No. 417A
7.	Specific impulse (I_{sp}) calc:		lb-sec/lb		

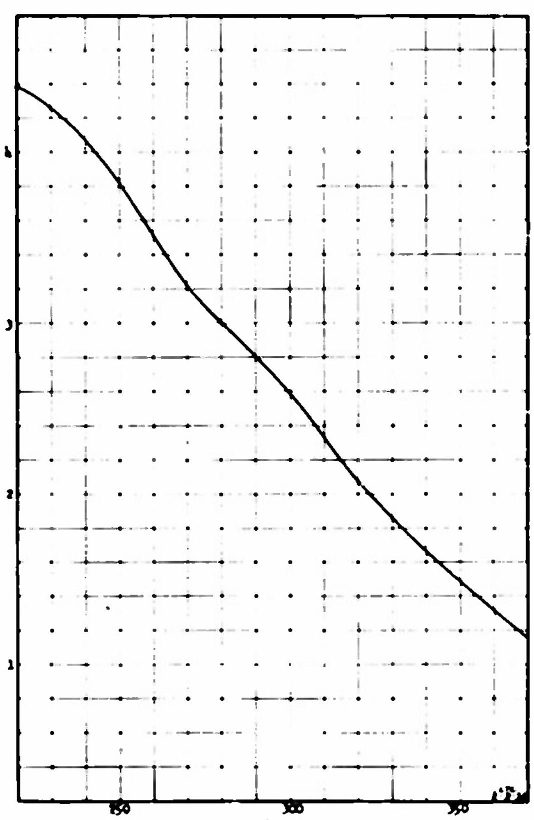
8. Physical form of compound (viscous liquid, crystalline type, etc.) White crystals

9. Simple microscope analysis data: (crystal studies)

10. Density (Macro method) 1.84 gm/cm³. (Micro or other method) ______ gm/cm³. (Explain on separate sheet any unique methods you use.)

11. Index of refraction: ($n_D^{25°C.}$) ______ 12. Color white 13. Odor none.

14. pH at 25°C. 2.3 (Method reference OSRD 3401 p.4, or OSRD 5968. Indicate method used, i.e. solvent and concentrations used, pH indicator paper or Beckman pH meter.) with Beckman pH meter in 0.011M acetone/water (25/1 volume ratio)



1,1,1,3,6,6,9,11,11,11-Decanitro-3,9-diaza-undecane in Methanol

17. Boiling point, or decomposition temperature: decomposes on melting °C. (underline which temperature is reported)

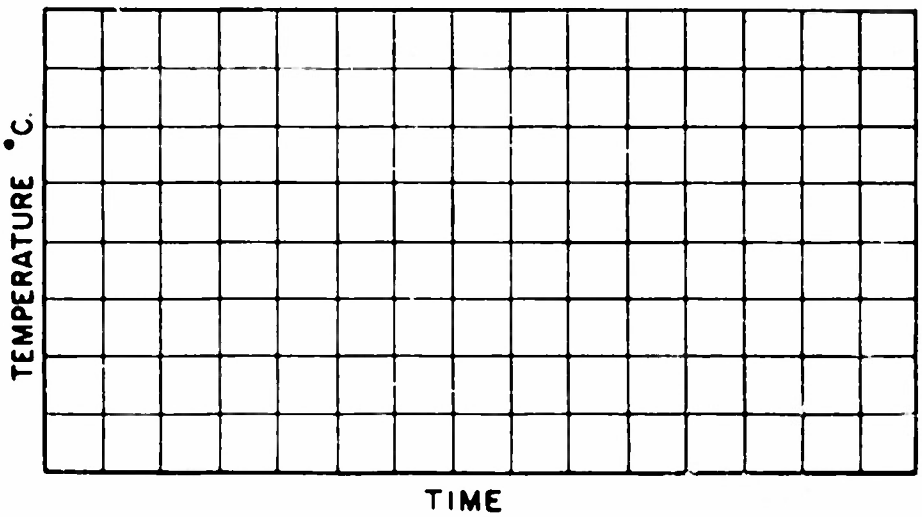
18. Heat of Vaporization: ______ g-cal./gm ______ BTU/lb.

19. Heat of Fusion: ______ g-cal./gm ______ BTU/lb.

20. Melting point: 170-175 °C. (d)

Report No. 682
SPIA/M3

21. Freezing point vs. Time Curve: (Please mark the coordinates with scale values that apply to the compound under study.)

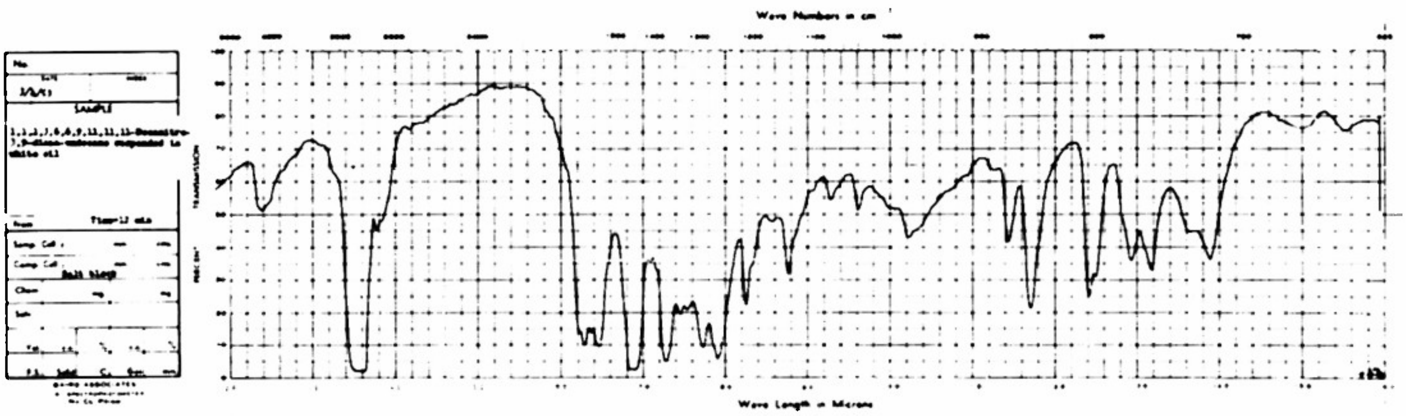


22. Solubility of new compound:

<0.1 g/100 ml H_2O at 25°C. ______ g/100 ml H_2O at ______ °C.

<0.1 g/100 ml toluene (name material used as solvent) at 25 °C.

50 g/100 ml acetone (name material used as solvent) at 25 °C.



1,1,1,3,6,6,9,11,11,11-Decanitro-3,9-diaza-undecane Suspended in White Oil
Time=12 min

Report No. 682
SPIA/MS

Under compatability we are considering the ability of two compounds to be in intimate contact (Note OSRD 5758 p. 21-22) over a long period of time without adverse effects on either the chemical or physical properties of either material. These tests will probably be of varied extent. They might be some of the following: (a) Standard stability tests. (b) Simple observations of exudation or separation at ambient or accelerated temperature. (c) Prepare thin sheets (.025" thick, 1" square) of the plasticized material. Separate the sheets with strips of cigarette paper or carbon paper and compress the stack in a "C" clamp. After several days note the oily collection on the paper. Please give reference to or describe procedure used. Item 28 is to indicate results when the compound is in contact with some material other than ethyl cellulose, nitrocellulose or rubber.

25. Compatability with ethyl cellulose: __________

26. Compatability with nitrocellulose: __________

27. Compatability with rubber: __________

28. Compatability with __________: __________

29. Polymerizing properties of the new compound:
    (a) By itself __________
    (b) In mixtures (with additives) __________
    (c) Inhibiting action on polymerization of:
        Thiokol __________
        Methacrylate __________
        Other compounds __________

30. Availability
    a. Amount now available? Research quantities
    b. When was available material first prepared? __________
    c. Amount prepared at that time? __________
    d. Is large production feasible? __________
    e. Plant capacity in existance, lbs/day? __________
    f. Outline steps for a quantity production method __________

31. Additional information: (toxicity, hazards, deterioration, oxygen balance, detonation rate, explosive power, ease of hydrolysis by water, etc. List references, reports, data books, etc. that refer to the compound.)

Calculated Lead Block Value = 177
Calculated Ballistic Mortar Value = 155
} Method of Aerojet Report No. 512, p. 8

Report No. 682
SPIA/M3

Data Questionnaire on

COMPOUNDS FOR USE AS INGREDIENTS OF PROPELLANTS AND OTHER EXPLOSIVES

Listed below are the characteristics which are believed to be of importance in the study of a compound which may possibly be used as a constituent of solid propellants or other explosives. It is not expected that all this information will be available for every compound. For example, with a compound showing poor stability, it is probably not advisable to investigate that compound farther. After placing such information as is available for a compound which you have prepared or tested on one of these forms, send it to SOLID PROPELLANT INFORMATION AGENCY, APL/JHU, 8621 Georgia Avenue, Silver Spring, Maryland. As additional information on the same or new compounds accrues, forward it on a similar form at a later date. The information submitted on these forms will be rewritten and published by SPIA in loose-leaf manual form. These forms may also be used as work or data sheets for your experimental studies. Extra copies are available upon request from SPIA. Suggestions for improvement of these forms are invited. If insufficient space has been provided for any item, attach separate sheets.

COMPOUND: 1,1,1,3,5,7,7,7-Octanitro-3,5-
Name diaza-heptane
Empirical formula $C_5H_6N_{10}O_{16}$
Structure: (configuration)

$$CH_2-\left[\begin{array}{l} NO_2 \\ | \\ NCH_2C(NO_2)_3 \end{array}\right]_2$$

Information submitted by:
Activity Aerojet Engineering Corporation
Person M.B. Frankel and L.T. Carleton
Date 6 February 1953

$$CH_2[NH_2 \cdot HCl]_2 + 2HOCH_2C(NO_2)_3 + 2NaOCOCH_3 \longrightarrow CH_2-\left[\begin{array}{l} H \\ | \\ NCH_2C(NO_2)_3 \end{array}\right]_2 \xrightarrow[Ac_2O]{HNO_3} CH_2-\left[\begin{array}{l} NO_2 \\ | \\ NCH_2C(NO_2)_3 \end{array}\right]_2$$

Preparation reaction(s):

1. Quantitative analysis: (% by weight)

	Carbon	Hydrogen	Oxygen	Nitrogen		
Calculated from formula	12.99	1.31	55.39	30.31		
By determination	13.44	1.23		30.45		

2. Burning properties: (compared to nitrocellulose, under nitrogen at atmospheric pressure:)

(faster? slower? residue? etc.)

3. Stability and Sensitivity: Plot any graphs on separate sheet

Discuss methods used when they vary from references. Give temperature used. (Use separate sheet if necessary.)

	Name of test	Recommended method	
a.	Impact Sensitivity	NOL ~~OSRD [illegible]~~	2.5-kg weight
b.	Thermal Stability	OSRD 3401 p.8	
c.	Vacuum Stability	NOL ~~OSRD [illegible]~~	48 hr at 100°C
d.	Temperature of Explosion	OSRD 3401 p.6	
e.	Temperature of Ignition	OSRD 3401 p.6	
f.	Impact Stability	Bureau of Mines Bull. No. 346, p. 72 (2-kg weight)	
g.			
h.			

RESULTS OF ABOVE TESTS

	Reference compound (designation-TNT, Tetryl, N.C., etc.)	New Compound test results
a.	Tetryl, 32 cm/2.5 kg	5.0 cm/2.5 kg
b.		
c.		>30 cc/g in 2 hr
d.		
e.		
f.	RDX, 30 cm/2 kg	5-10 cm/2 kg
g.		
h.		

4. Heat of formation: (ΔH) ± −18 (indicate sign) Kg. calories at 25°C., 1 atm. pressure

Report No. 682
SPIA/M3

	By Experiment		By Calculation	Method (Description or reference. Separate sheet if necessary.)
5. Energy of explosion (Q) (at 25°C. H_2O liquid)		cal/gm		
6. Heat of combustion (H_c) (at 25°C. H_2O liquid)	1423	cal/gm	1427	Aerojet Report No. 417A
7. Specific impulse (I_{sp}) calc:		lb-sec/lb		

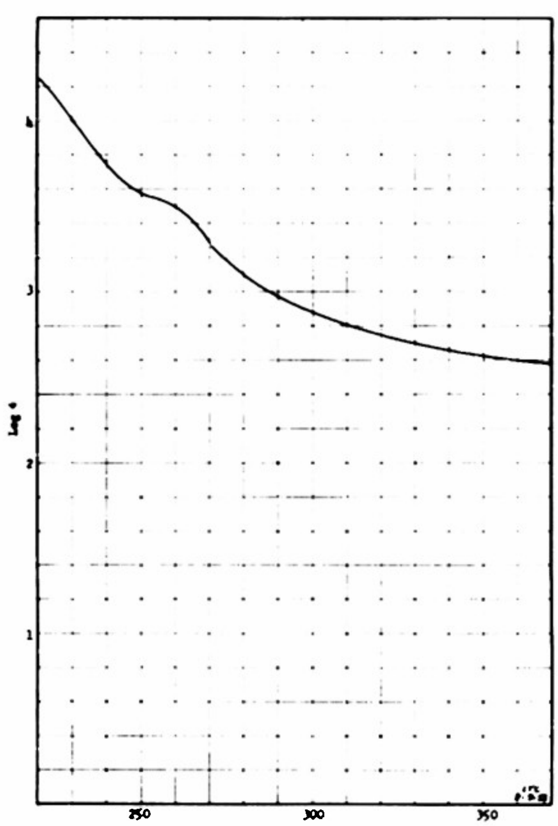
8. Physical form of compound (viscous liquid, crystalline type, etc.) Flaky white crystals

9. Simple microscope analysis data: (crystal studies)

10. Density (Macro method) 1.87 gm/cm³. (Micro or other method) ______ gm/cm³. (Explain on separate sheet any unique methods you use.)

11. Index of refraction: ($n_D^{25°C.}$) ______ 12. Color white 13. Odor none.

14. pH at 25°C. 3.8, decreasing quickly (Method reference OSRD 3401 p.4, or OSRD 5968. Indicate method used, i.e. solvent and concentrations used. pH indicator paper or Beckman pH meter.) with Beckman pH meter in 0.014M acetone/water (5/1 volume ratio)



1,1,1,3,5,7,7,7-Octanitro-3,5-diaza-heptane in Ethanol

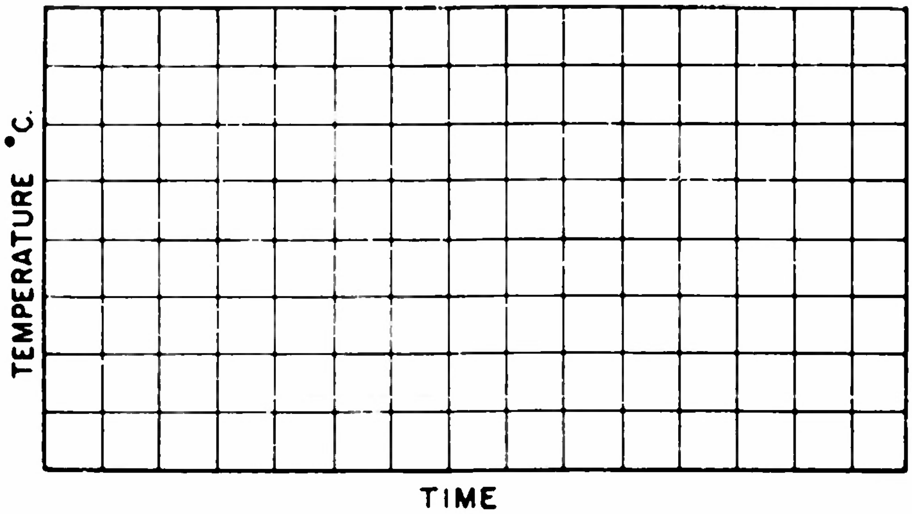
17. Boiling point, or decomposition temperature: ______ °C. (underline which temperature is reported)

18. Heat of Vaporization: ______ g-cal./gm ______ BTU/lb.

19. Heat of Fusion: ______ g-cal./gm ______ BTU/lb.

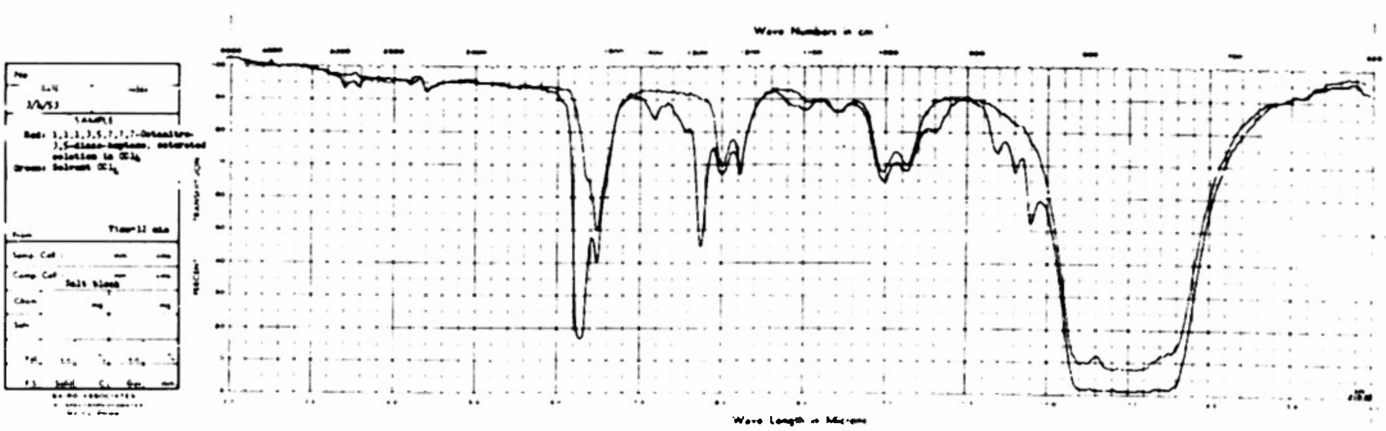
20. Melting point 84-85 °C.

21. Freezing point vs. Time Curve: (Please mark the coordinates with scale values that apply to the compound under study.)



22. Solubility of new compound:

<0.1	g/100 ml H_2O at 25°C.		g/100 ml H_2O at	°C.
42	g/100 ml	toluene (name material used as solvent)	at 25	°C.
480	g/100 ml	acetone (name material used as solvent)	at 25	°C.



Red: 1,1,1,3,5,7,7,7-Octanitro-3,5-diaza-heptane, Saturated Solution in CCl_4
Green: Solvent CCl_4
Time=12 min

Under compatability we are considering the ability of two compounds to be in intimate contact (See OSRD 5758 p. 21-22) over a long period of time without adverse effects on either the chemical or physical properties of either material. These tests will probably be of varied extent. They might be any of the following: (a) Standard stability tests. (b) Simple observations of exudation or separation at ambient or accelerated temperature. (c) Prepare thin sheets (.025" thick, 1" square) of the pl[illegible] material. Separate the sheets with strips of cigarette paper or carbon paper and compress the [illegible] a "C" clamp. After several days note the oily collection on the paper. Please give reference or describe procedure used. Item 28 is to indicate results when the compound is in contact with a material other than ethyl cellulose, nitrocellulose or rubber.

25. Compatability with ethyl cellulose: ______

26. Compatability with nitrocellulose: ______

27. Compatability with rubber: ______

28. Compatability with ______ : ______

29. Polymerizing properties of the new compound:
(a) By itself ______
(b) In mixtures (with additives) ______
(c) Inhibiting action on polymerization of:
Thiokol ______
Methacrylate ______
Other compounds ______

30. Availability
a. Amount now available? Research quantities
b. When was available material first prepared? ______
c. Amount prepared at that time? ______
d. Is large production feasible? ______
e. Plant capacity in existance, lbs/day? ______
f. Outline steps for a quantity production method ______

31. Additional information (toxicity, hazards, deterioration, oxygen balance, detonation rate, explosive power, ease of hydrolysis by water, etc. List references, reports, data books, etc. that refer to the compound.)

Oxygen Balance = +10.4

When adjusted to zero oxygen balance with 12.3% TNT, the calculated lead block value is 196 and the calculated ballistic mortar value is [illegible]6.

(Method of Aerojet Report No. 512, p. 8.)